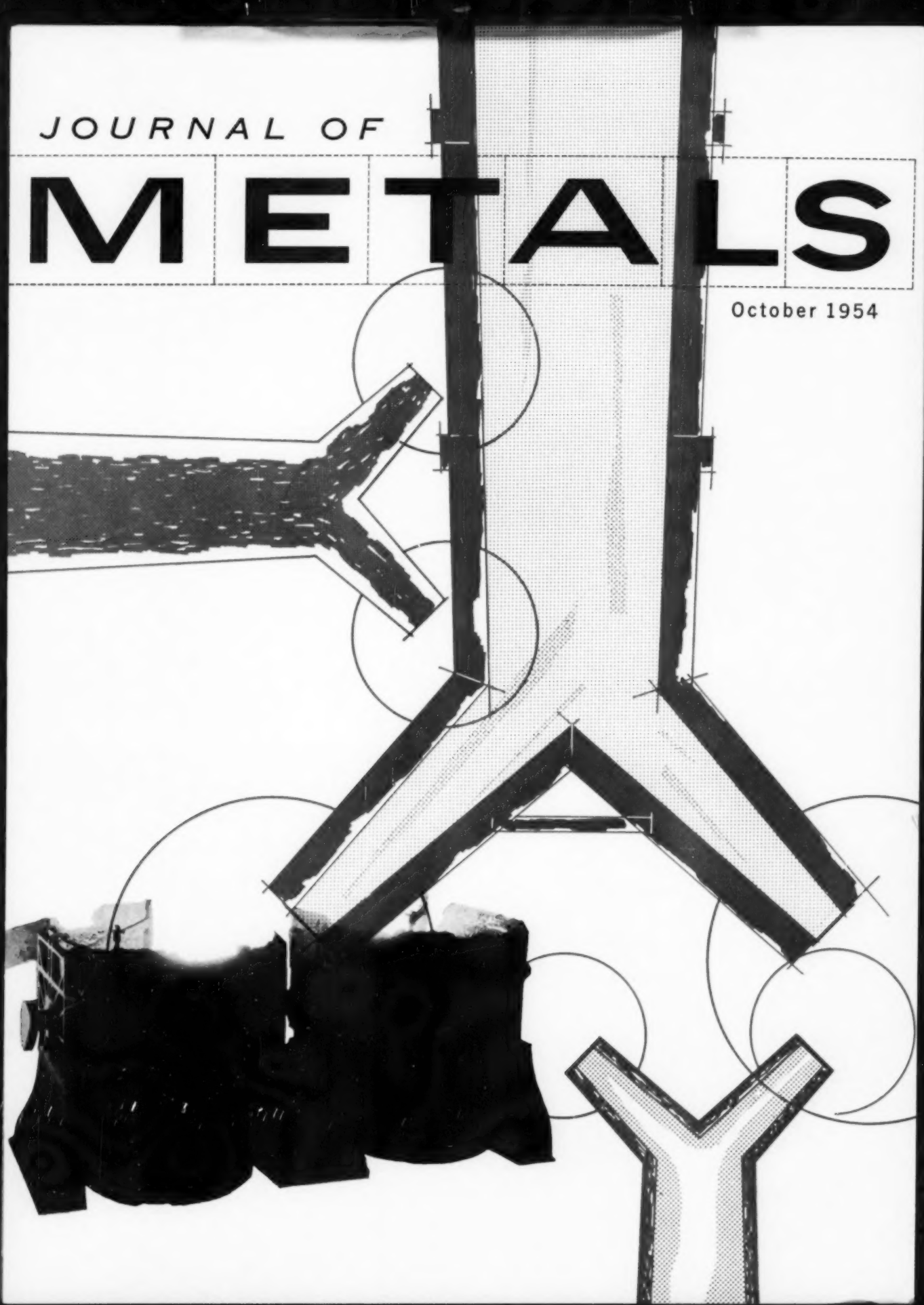
JOURNAL OF

METALS

October 1954

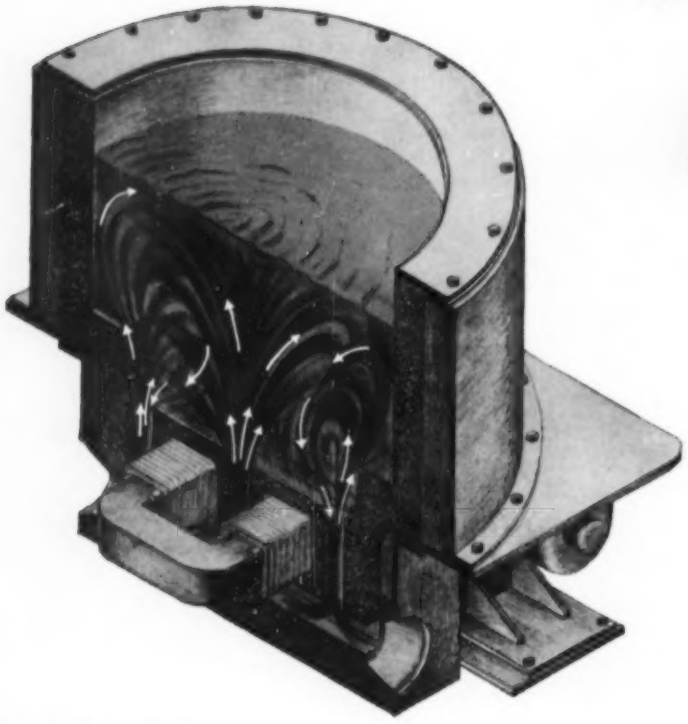


The Furnace that Stirs Itself!

ELECTROMAGNETIC PRESSURE CIRCULATES MOLTEN METAL

— In this 60 Cycle —

AJAX TAMA-WYATT *Induction Furnace*



Heat is generated only in the melting channels. Controlled stirring *(neither too much nor too little)* guarantees uniformity of metal temperature and alloy composition and also leads to efficient melting of light scrap. Tiresome puddling is eliminated. The metal is held entirely in an inert refractory lining. The atmosphere is cool and free from contaminating gases.

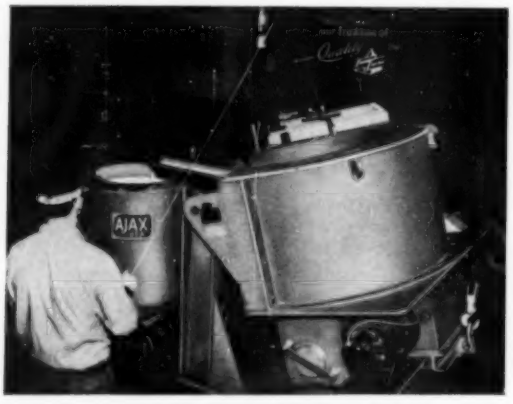
No Other Method Enables such Completely CONTROLLED MELTING

Today, AJAX builds a complete line of these time-tested furnaces in standard sizes up to 333 kW for the dependable melting of aluminum, brass, copper and zinc. Units for special applications are carefully engineered to specifications.

TWIN COIL INDUCTORS

Lead Non-Ferrous Melting

The sectional view above shows the twin coil stirring action of the 100 kW Ajax-Tama Wyatt 60 cycle induction furnace. Heat induced in the secondary channels below is conveyed throughout the melt by electromagnetic circulation as shown by the arrows. The 100 kW furnace shown here, which will be on display at the 1954 Metal Show, is one of a family of twin coil furnaces available today for melting rates from 300 to 10,000 lbs. per hour.



We cordially invite you to inspect the 100 kW AJAX furnace exhibited at the NATIONAL METAL SHOW, Chicago, Nov. 1-5, 1954, Booth #752.

AJAX TAMA-WYATT

AJAX ENGINEERING CORP., TRENTON 7, N. J.

INDUCTION MELTING FURNACE

AJAX ELECTRO METALLURGICAL CORP., and Associated Companies
AJAX ELECTROTHERMIC CORP., Ajax Northrup High Frequency Induction Furnaces
AJAX ELECTRIC CO., INC., The Ajax Multgren Electric Salt Bath Furnace
AJAX ELECTRIC FURNACE CORP., Ajax Wyatt Induction Furnaces for Melting

JOURNAL OF

METALS

VOL. 6, NO. 10 OCTOBER, 1954

Published to provide a continuing, authoritative, and up-to-date record of technological, engineering, and economic progress in all branches of the metals industry by the Metals Branch American Institute of Mining and Metallurgical Engineers, Inc. 29 West 39th Street, New York 18

• • •

ALVIN S. COHAN
Manager of Publications
Editor, JOURNAL OF METALS
EDWIN M. WEISS
Associate Editor

• • •

M. A. MATZKIN M. SNEDEKER
G. M. ZILL
Assistant Editors

GRACE PUGSLEY Production Manager
W. J. SEWING Asst. Prod. Mgr.

• • •

METALS BRANCH COUNCIL

R. R. McNaughton Chairman
E. O. Kirkendall Secretary
J. H. Scaff IMD Chairman
J. S. Marsh ISD Chairman
P. T. Stroup EMD Chairman
M. Cohen R. D. Parks
T. J. McLaughlin D. S. Eppelsheimer

EDITORIAL ADVISORY COMMITTEE

J. S. Smart, Jr. Chairman
W. A. Dean Evan Just
J. C. Kinnear, Jr. John Marsh
T. D. Jones M. Tenenbaum

TECHNICAL PUBLICATIONS COMMITTEES

Robert Aborn AIME Chairman
Otto Zmeskal ISD Chairman
S. J. Dickinson EMD Chairman
W. R. Hibbard, Jr. IMD Chairman
E. J. Kennedy, Jr. Secretary

• • •

FRED STANLEY
Eastern Advertising Manager
BOB WILSON
Western Advertising Manager
84 E. Randolph St., Chicago

• • •

The AIME Publishes:
Journal of Metals
Mining Engineering
Journal of Petroleum Technology

COVER

Double heats posed the problem of handling the increased steel tonnage with existing open hearth pit facilities. The symposium beginning on p. 1119 describes tapping with bifurcated spouts into two ladles. Harry Brocke's cover this month illustrates this latest innovation in open hearth practice.

NEWS

Meet the Authors	1096	Drift of Things	1109
Employment Service	1100	Journal of Metals Reporter	1115
Books for Engineers	1102	AIME News	1163
Industrial Notes	1103	Personals	1167
Trends	1104	Coming Events	1170

TECHNICAL FEATURES

Hot Pressing, Press Forming Loom As Answers to Titanium Fabrication — H. W. Dodds, O. F. Davies — 1116

Bifurcated Spouts Increase Open Hearth Output—Close Supervision Prevents Tieups — C. N. Straney — 1119

Increased Shop Tonnages Realized — E. C. Sorrells — 1120

Double Heats Show No Decrease in Quality — J. H. Kelley — 1121

Brick Sleeve Solves Tapping Problem — H. H. Northrup — 1123

Transportation Factors Are Key to Development of Caribbean Bauxite For U. S. Aluminum Industry — A. F. Johnson — 1124

Fuel Balance at Steubenville Works Controlled by Utilizing Summarizers — J. H. Dickson, G. C. Nuzum — 1127

It Took the Mesabi Discovery To Beat Edison's Taconite Venture — 1130

TRANSACTIONS

Methods for Separating Rare-Earth Elements in Quantity as Developed at Iowa State College — F. H. Spedding, J. E. Powell — 1131

Slag-Metal-Graphite Reactions and the Activity of Silica in Lime-Alumina-Silica Slags — J. C. Fulton, J. Chipman — 1136

Phase Transformations in Titanium-Rich Alloys of Iron and Titanium. — D. H. Polonis, J. G. Parr — 1148

Quantitative Substructure and Tensile-Property Investigations of Nickel Alloys — B. Ancker, E. R. Parker — 1155

TECHNICAL NOTES

Influence of Boron on Hardenability of Steel — J. C. Fisher — 1146

Published the first day of each month by the American Institute of Mining and Metallurgical Engineers, Inc., 29 West 39th Street, New York 18, N. Y. Telephone: PEnnsylvania 6-9220. Subscription $8 per year for non-AIME members in United States and North, South and Central America; $10, foreign; $6 for AIME members, or $4 additional in combination with a subscription to "Mining Engineering" or "Journal of Petroleum Technology." Single copies, 75 cents; single copies foreign, $1.00; special issues, $1.50 . . . The AIME is not responsible for any statement made or opinion expressed in its publications . . . Copyright 1954 by the American Institute of Mining and Metallurgical Engineers, Inc. . . . Registered cable address, AIME New York . . . Indexed in Engineering Index, Industrial Arts Index and Chemical Abstracts . . . Reentered as second-class matter Sept. 21, 1949, at the post office at New York, N. Y., under the Act of March 3, 1879. Additional entry established at Manchester, N. H.

For maximum efficiency in the production of specimens in the metallurgical laboratory the Buehler cabinet type polishing table with companion storage cabinets represents the latest modern development of this type of equipment.

The convenience of this streamlined polishing equipment saves time and encourages the operator to produce the highest quality of polished sample.

Item No. 1511 is a two-unit polishing table with Formica top approximately 60" long x 27" deep by 30" high to table top. Two 12" swing spouts, drain, 8" diameter wash bowl, plumbing and wiring.

Recommended accessories to complete an efficient set up for maximum convenience are: No. 1512 storage cabinet with recessed light and No. 1513 supporting panel for installation above polishing desk. Or, No. 1514 floor model storage cabinet. Both these cabinets can be used together to advantage in most laboratories.

The Formica top and back on the table and cabinet is installed with a smooth Formica edge that eliminates all metal rims that may form pockets for water and dirt. Covers are held in place on the back by magnetic holders. The large 8" wash bowl is a new feature that enables the operator to use both hands in washing specimens.

All metal construction finished in hammer tone grey makes a very attractive appearance. Prompt delivery can be made on these new items.

The Buehler Line of Specimen Preparation Equipment Includes . . . Cut-Off Machines • Specimen Mount Presses • Power Grinders • Emery Paper Grinders • Hand Grinders • Belt Surfacers • Mechanical and Electro Polishers • Polishing Cloths • Polishing Abrasives

Buehler Ltd. METALLURGICAL APPARATUS
2120 Greenwood Avenue, Evanston, Illinois

Meet The Authors

John Dickson (p. 1127) is a graduate of Pennsylvania State University (B.S., 1950). He joined Wheeling Steel Corp. after graduation and is presently a field engineer in the utilities dept., Steubenville, Ohio.

J. DICKSON

J. G. PARR

J. Gordon Parr (p. 1148) has been a research associate on the staff at the University of British Columbia since 1953. Born at Peterborough, England, Dr. Parr received his B.Sc. from Leeds University and his Ph.D. from Liverpool University. From 1947 to 1948 he was a research assistant at Leeds and then became a lecturer in metallurgy at Liverpool University. He was the recipient of Nuffield Foundation traveling scholarships in 1947 and 1951.

Betsy Ancker (p. 1155) is research physicist with the minerals research laboratory and a lecturer in the physics dept., University of Berkeley. Dr. Ancker was born at St. Louis and is a graduate of Wellesley College and University of Tuebingen, Germany.

Grover Nuzum (p. 1127) began his career with Wheeling Steel Corp. in instrument repair. In 1947 he was made a field engineer assigned to the open hearth and is presently in that capacity at the Steubenville Works.

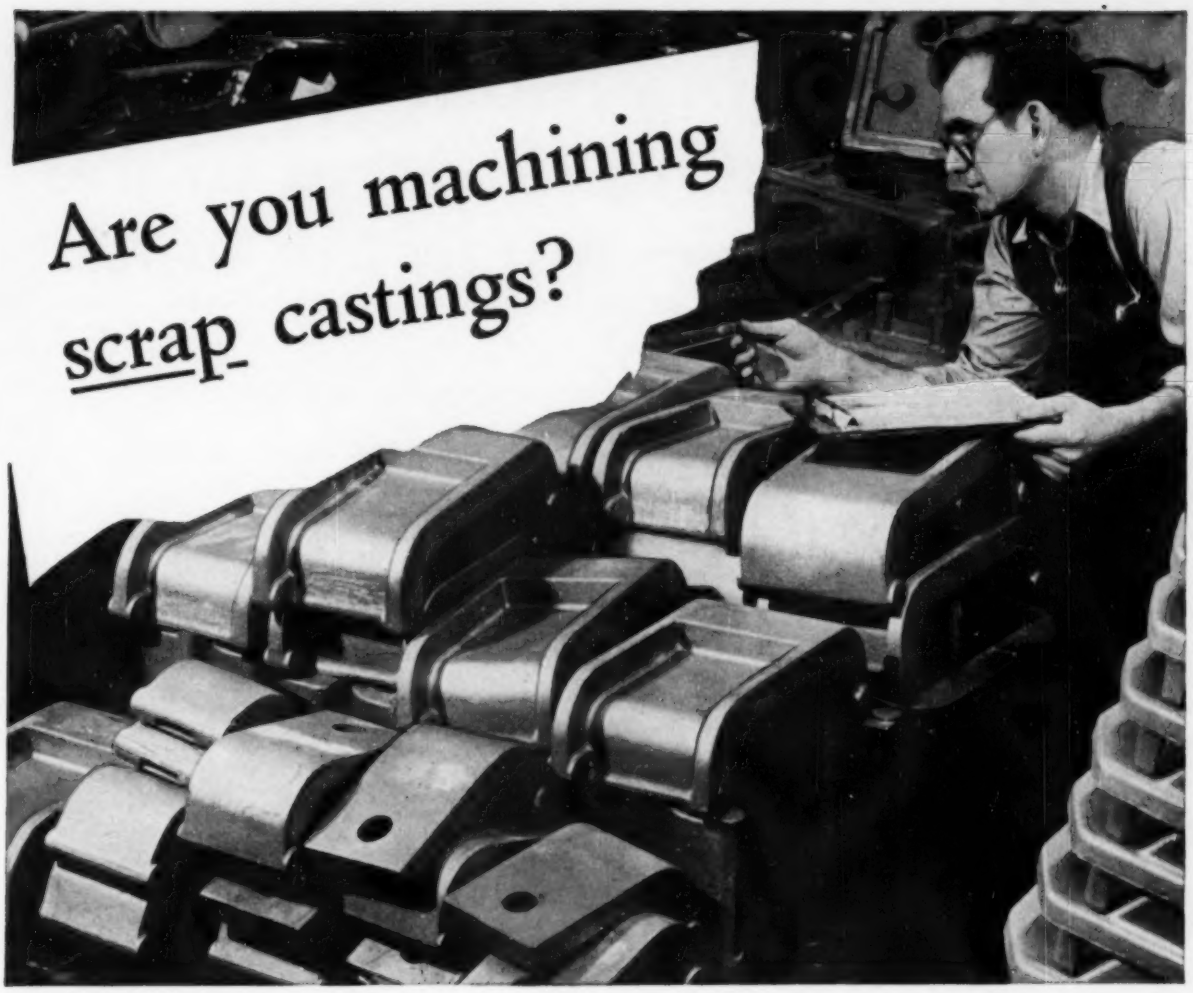
G. NUZUM

E. C. SORRELLS

E. C. Sorrells (p. 1120) has been with the U. S. Steel Corp. 18 years. He has worked as furnace operator, metallurgical observer, metallurgical foreman, shop superintendent, assistant superintendent, and assistant divisional superintendent. He is presently division superintendent, steel production at the Gary Works. He previously presented a paper before AIME in 1944.

J. H. Keiley (p. 1121) assistant superintendent of open hearth and Bessemer dept., Sparrows Point plant of the Bethlehem Steel Co., has been with the company since his graduation from Pennsylvania State University in 1935. His first position was in the metallurgical dept. at the Johnstown plant. From there he moved to Bethlehem in 1937. Since 1941 he has been foreman and then assistant superintendent in the open hearth dept.

Arthur F. Johnson (p. 1124) was born at Boulder, Colo., and graduated from the University of Colorado and Massachusetts Institute of Technology. From 1926 to 1933 he was associated with the Aluminum Co. of America. He did consulting work for several years, and in 1936 joined the American Smelting & Refining Co. Mr. Johnson became associated with the Reynolds Metals Co. in 1949 and joined the Olin Industries in 1953. Mr. Johnson is a Member of AIME.



Non-destructive inspection with a G-E x-ray unit insures a better product at lower cost

IF YOU'RE A USER OF CASTINGS, there's no need to buy blindly. With the all-seeing eye of x-ray, you can spot internal defects *before* costly machining. Even those deep-seated flaws that would not show up during machining can be eliminated as a source of potential failure of your product.

If you sell castings, on the other hand, x-ray inspection can protect your reputation — save your customers money and aggravation — give you a "plus" sales feature. In many foundries, x-ray is also used as a development tool — to disclose where savings can be made, designs improved, new techniques developed.

The possibilities for the profitable application of x-ray are as broad as industry itself. In addition to castings — weldments, intricate assemblies, the composition of the materials themselves can be checked.

Your G-E x-ray representative will be glad to analyze your requirements. With General Electric's line of 25 models, he can recommend the unit best suited to your needs. Call him today, or write X-Ray Department, General Electric Company, Milwaukee 1, Wis., Rm. AY64

General Electric x-ray apparatus can be yours . . . without initial capital investment . . . on the G-E Maxiservice® rental plan

Progress is our most important product

GENERAL ELECTRIC

"Today, the most fruitful prospecting is done in the laboratory—not in the field.

Develop a process to suit the ore,"

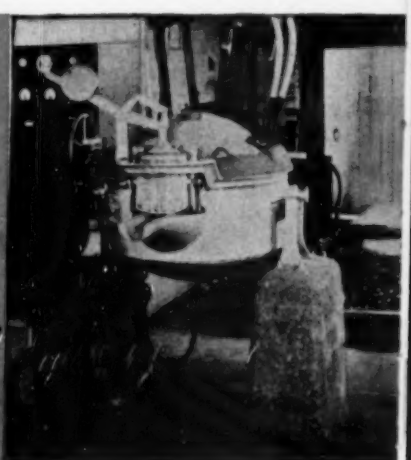
say Messrs. P. B. Dettmer, General Superintendent, and L. J. Lichty, Managing Director, of Cobalt Chemicals Limited, Cobalt, Ont.

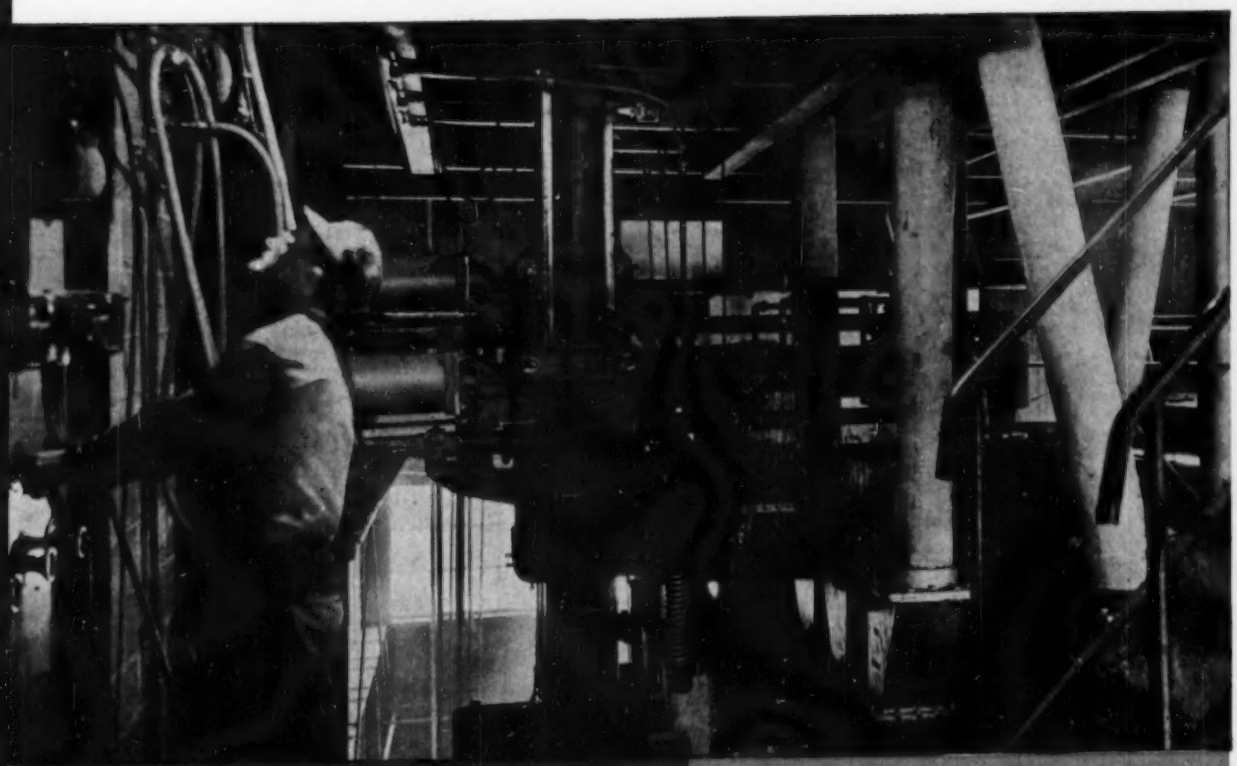
Quebec Metallurgical Industries, Ltd. followed exactly this policy in directing development work for Cobalt Chemicals Limited. Research and pilot plant operations were carried on at their Ottawa Laboratories. They resulted in establishment of the process now employed on a commercial basis for the treatment of arsenical silver-cobalt-nickel concentrates produced in the Cobalt area.

The 600 KVA. Smelter, shown on the opposite page, is now operating on a commercial basis at Cobalt Chemicals Limited. Concentrates, mixed with fluxes, are put through this furnace and smelted to matte; arsenious oxide is also produced as a valuable side product. The matte is ground, roasted, sulphated and leached, after which copper, nickel, cobalt and silver are selectively precipitated and reduced to metallic form.

The "work horse" during the initial experiments, when their chemists were formulating the process, was the 50 K.W. Lectromelt* Furnace. Although unimpressive in appearance, it has the flexibility which enables researchers to proceed quickly and surely. It permits them to make a quick shift as a new approach is suggested.

The pilot plant which made possible the next step —taking the now proven process out of the experimental stage toward production— was this 100 K.W. Lectromelt Furnace. Its larger size and higher power let their metallurgists approximate conditions expected under actual smelting conditions. Thus fewer faults remained to be corrected when operations moved from the Laboratories at Ottawa into the plant at Cobalt.

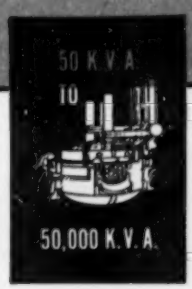




Lectromelt electrode holders and controls for the 600 KVA. smelting furnace of Cobalt Chemicals Limited. Concentrates are charged through the ducts seen at the right.

Left: Exacting control of the furnace produces a molten slag and matte at tapping time. At this stage, the matte contains cobalt, silver, nickel, copper and sulfur.

Whether your smelting process is still experimental or ready to put into production, there's Lectromelt equipment able to serve you. Our engineers have been conducting continuing research for many years on electrothermic reductions. This experience is available to you. For Catalog No. 105, write Pittsburgh Lectromelt Furnace Corporation, 326 32nd St., Pittsburgh 30, Pa.



®REG. T. M. U. S. PAT. OFF.

MOORE RAPID

WHEN YOU MELT... *Lectromelt*

— Personnel Service —

THE following employment items are made available to AIME members on a nonprofit basis by the Engineering Societies Personnel Service Inc., operating in cooperation with the Four Founder Societies. Local offices of the Personnel Service are at 8 W. 40th St., **New York 18;** 100 Farnsworth Ave., **Detroit;** 57 Post St., **San Francisco;** 84 E. Randolph St., **Chicago 1.** Applicants should address all mail to the proper key numbers in care of the New York office and include 6c in stamps for forwarding and returning application. The applicant agrees, if placed in a position by means of the Service, to pay the placement fee listed by the Service. AIME members may secure a weekly bulletin of positions available for $3.50 a quarter, $12 a year.

MEN AVAILABLE

Metallurgical Engineering Executive, age 52. Broad management and engineering experience with large and small steel companies. Planned, organized and installed projects in U.S. and overseas. Desires responsible management or consulting position. Free to travel. Location immaterial. Excellent health. M-128.

POSITIONS OPEN

Metallurgist, 40 to 45, with at least 10 years ferrous and nonferrous experience including precision casting. Salary, $10,000 a year. Location, Brooklyn, N. Y. W412.

Research Powdered Metallurgist, graduate or equivalent, with laboratory and plant experience. Will consider recent graduate who has had powdered metal studies. Must be able to make metallographic laboratory studies, develop holding media and engage in production control. Work with diamond, abrasives and base media in a sintering and adhesion process for a manufacturer of diamond saws for nonmetallic cutting. $6000 maximum and $4200 for recent graduate. Location, Los Angeles, Calif. S883.

Plant Manager, 30 to 40, with metal stamping, polishing and plating experience to supervise operations for small specialty sheet metal products manufacturer. Salary, $6000 to $8000 a year plus bonus. Location, Queens, N. Y. W383.

Welding Engineer, metallurgical graduate, with at least eight years structural, plate and sheet arc welding experience. Salary, $7200 to $8400 a year. Location, Pennsylvania. W400(a).

Mill Foreman, young, graduate in ore dressing, experienced in screens, jigs, Humphreys spiral, air-table and magnetic separation for columbite-tantalite mill. Must be single. Salary, $4800 a year. Location, South America. F408.

Product Engineer, with milling and marketing experience in nonmetallics field for rock products development and sales promotion work. Salary, $10,000 to $12,000 a year. Location, California. W427.

Production Engineer, metallurgical or chemcial, for powdered metals division; must have experience in mass production brazing of precision parts in a continuous atmosphere controlled furnace and have a knowledge and experience of fixtures, gas crackers, both endothermic, exothermic, and dissociated ammonia and temperature controlled devices, recorders and thermocouples. Location, Midwest. W348.

Metallurgical Engineer, with experience in mineral dressing or process metallurgy for uranium research and development on various types of ores. Salary open. Location, Massachusetts. W416.

Metallurgist, graduate preferred, with 2 to 3 years experience in foundry, including quality control, advise mixes. Salary, $6000 a year. Location, upper New York state. W243.

Mechanical or Metallurgical Engineer, young, Ph.D. in M.E., with a strong major in metallurgical engineering, for a ballistics laboratory. Location, Maryland. W161.

process metallurgist

to process-engineer non-ferrous reduction plants

The work is basically economic evaluation of metal processes, process-design of new plants, and technical assistance to sales. The man we're looking for has a knowledge of reduction-processes for such metals as nickel, copper, aluminum, or titanium.

We prefer someone with the potential to lead other process-engineers. Please send us your resume, attention Personnel Department. We'll respect your confidence.

C. F. BRAUN & CO.

Engineers · Consultants · Constructors
Serving the Refining Industries Since 1909

ALHAMBRA, CALIFORNIA

Sales Manager, with at least 10 years experience in supervising metal products sales covering stampings and fabricated sheet steel products. Salary, $12,000 to $15,000 a year plus bonus. Location, New England. W401(b).

Instructor or Assistant Professor, to assist in metallography laboratory, serve as substitute instructor for physical metallurgy courses. Will teach iron and steel production first semester, and nonferrous production metallurgy, second semester. Salary dependent on applicant's education, training, and experience. Location, West. W131.

Project Engineer with nonferrous experience for research in copper powder, electrodeposition, atomization, reduction. Salary, $4420 to $5460 a year. Location, New Jersey. Y9849.

Sales Engineer, 30 to 45, metallurgical engineering degree, with industrial sales experience. Will sell electrical contacts, thermo-metals, powder metals and special purpose alloys. Salary, $5200 to $7200 a year. Location, Newark, N. J. area, and later assignment in East or Midwest. W213.

Metallurgist to act as associate consultant. Must have considerable and varied experience in the field of nonferrous metals, with the accent on hydro rather than pyrometallurgy. Will be required to make trips overseas. Salary open. Headquarters, New York, N. Y. W444.

Metallurgical Engineer, to take charge of the metallurgical problems of a mining operation. He should have a thorough knowledge of flotation, cyanidation, lixiviation and, if possible, have some knowledge of running a sulphuric acid plant. Accommodations for man and wife only. Salary open. Location, Chile. F172.

METALLURGIST-PHYSICIST
PERMANENT MAGNETS

Experienced in Alnico production and control with practical and theoretical background for developing magnetic material. Must be U. S. citizen. Location New Jersey.

Box J-20M AIME
29 West 39th St., New York

METALLURGIST AVAILABLE—Physical metallurgist with strong background in metallurgical engineering seeks change in position. Well grounded in fabrication, processing and property studies of commercial nonferrous—ferrous metals, zirconium, titanium, hi-temperature, rare earth metals. 15 years diversified experience in heavy industry, automotive and atomic energy field including 7 years on nuclear reactors. Inclination toward broad field of metallurgical activities. Supervisory and staff duties. Adaptable, cost conscious researcher with mature and tactful judgment. Thorough laboratory experience. M.S.-Ph.D., linguist, age 40. Interested in responsible research planning, coordination or technical administration.

Box J-21M AIME
29 West 39th St., New York 18

OPERATOR OF POWDERED METAL DEPARTMENT

Here is a rare opportunity for a man qualified to operate a small powdered metal parts department.

He should have 5 years or more experience with technical background equivalent to a graduate engineer. His background should include tool & die experience with a thorough knowledge (precious metals) of fabrication and related metallurgy.

Must have ability to control quality & cost and recommend products to be manufactured. We want him to develop a small dept. into a significant part of our business.

Forward complete details of your experience, training and salary requirements. All replies will be treated confidentially.

Box J-18M AIME
29 West 39th St., New York 18

DEVELOPMENT ENGINEER

To develop techniques for non-destructive inspection of reactor components. An unusual opportunity to set up laboratory and direct experimental programs. Two years of applicable experience required. Laboratories located in suburban Pittsburgh near excellent housing of all types. Your application will be handled promptly and confidentially. Please write in detail giving complete information concerning training, experience and salary requirements to Mr. C. F. Stewart, Supervisor of Industrial Relations, P.O. Box 1468, Pittsburgh 30, Pennsylvania.

WESTINGHOUSE
ATOMIC POWER DIVISION

LIGHT METALS METALLURGIST

Ph.D., 44 yr., unusual broad background as manager of research and control laboratories, reduction plants, billet foundries, extrusion departments, tube and wire drawing departments.

Box J-19M AIME
29 W. 39th St., New York 18, N. Y.

RESEARCH PHYSICAL METALLURGISTS OR MECHANICAL ENGINEERS

Wanted by progressive company for research program on mechanics of materials, including fatigue, vibrations, creep and other elevated temperature properties; laboratory located in area of attractive surroundings and pleasant climate in Northwest. Positions to be filled are:

(a) Senior investigator capable of planning and administration of at least part of the above program. Should have equivalent of Ph.D. with some additional research experience. Demonstrated success in industrial research accepted as substitute for advanced degree.

(b) Junior investigator, recent college graduate with some additional research experience preferred but not essential.

Salaries depend upon experience and past record. Excellent opportunity for advancement. Replies will be held in strict confidence. Send full details of experience and salary requirements. Our people know of this advertisement.

Box J-16M AIME
29 West 39th St., New York 18, N. Y.

METALLURGIST: Several openings available in metallurgical department connected with stainless steel operations in Eastern section. B.S. degree in Engineering required. Excellent opportunity for young man to establish in large and progressive organization where employee training and development receive keenest attention to fulfill company policy of promotions within.

Box J-17M AIME
29 West 39th St., New York 18

Books for Engineers

ORDER YOUR BOOKS THROUGH AIME—Address Irene K. Sharp, Book Department. Ten per cent discount given whenever possible. Order Government publications direct from the agency concerned.

Fiberglas Reinforced Plastics, by Ralph H. Sonneborn and others. *Reinhold Publishing Corp.* $4.50, 240 pp., 1954.—Publishers claim that this is the first complete treatment ever published on reinforced plastics. The book is aimed at both designers and executives in the materials industries and covers in detail the resins and glass reinforcements used in reinforced plastics, molding techniques, inspection and testing, properties, and design considerations.

PROFESSIONAL SERVICES

Limited to AIME members, or to companies that have at least one AIME member on their staffs. Rates $40 per year per inch.

SCIENTISTS
CONSULTANTS
METALLURGISTS

Small Jobs Welcomed

SAM TOUR & CO., INC.

Laboratories and offices
44 Trinity Place, New York 6, N. Y.

Testing—Certifying
American Standards
Testing Bureau, Inc.

HANS NEUBERT

PRECISE PROMPT

TECHNICAL TRANSLATIONS FROM GERMAN, SPANISH, FRENCH INTO ENGLISH. $1.00 per 100 WORDS OF THE ENGLISH TRANSLATION.

31 Hilltop Ave. Clark-Rahway, N.J.

MAX STERN
Consulting Engineer

Expert for Scrap Recovery and Shipwrecking — Modernization of Plants and Yards for Ferrous and Nonferrous Metal Scrap

149 Broadway New York 6, N. Y.

H. L. TALBOT
Consulting Metallurgical Engineer

Extraction and Refining of Base Metals Specializing in Cobalt and Copper

Room 331, 84 State St., Boston 9, Mass.

Manual for Plastic Welding, Vol. II, Polyethylene, by G. Haim and J. A. Neumann. *Industrial Publishing Co.* $6.00, 1954.—The manual is confined to gas welding of plastics and its applications. Audience sought by the book are practical welders and engineers seeking to incorporate plastic materials into designs.

Plastics Engineering Handbook, Society of the Plastics Industry, Inc. *Reinhold Publishing Corp.* $15.00, 813 pp., 1954.—This is a completely rewritten and expanded version of the SPI Handbook. Complete chapters of all new material have been added, such as the one on tooling with plastics, which describes the newly-developed uses of plastics in production tools for the aeronautical, automotive, and other industries. Another innovation is the special fold-out adhesives chart found in the chapter dealing with cementing and assembly.

ASTM Specifications for Steel Piping Materials, prepared by ASTM Committee A-1 on Steel. *American Society for Testing Materials.* $3.75, 370 pp., 1954.—The 1954 edition of this compilation sponsored by ASTM Committee A-1 on Steel contains their latest approved form for the 53 widely used ASTM specifications for carbon steel and alloy steel pipe and tubing, including stainless. Materials covered include: pipe used to convey liquids, vapors and gases at normal and elevated temperatures; heat exchanger and condenser tubes; boiler, superheater, and miscellaneous tubes, and still tubes for refinery service. Specifications for materials used in pipe and related installations include: castings, forgings, and weld fittings; bolts and nuts.

S. S. Ocean Vulcan Static Experiments, Report No. R6. *Her Majesty's Stationery Office.* $6.75, 49 pp., and 107 diagrams, 1953.—The Admiralty Ship Welding Committee, in its study of the special problems arising from the use of welding in ships' structures, has carried out a program of investigations to compare the structural behavior of welded and riveted ships under loadings experienced in still water. Results are prepared in diagram form. Available from British Information Services, New York, N. Y.

S. S. Clan Alpine Static Experiments, Report No. R7. *Her Majesty's Stationery Office.* $6.75. 18 pp., and 116 diagrams, 1953.—This report also deals with riveted and welded construction in relation to stresses imposed upon an ocean vessel. Available from British Information Services, New York, N. Y.

S. S. Ocean Vulcan Sea Trials, Report No. R11. *Her Majesty's Stationery Office.* $6.75, 12 pp. and 124 diagrams. Similar to the previous two reports, but dealing with actual observations made while the ship was at sea. Available from British Information Services, New York, N. Y.

Bibliographic Survey of Corrosion. *National Assn. of Corrosion Engineers.* $12.50, 346 pp., 1954.—The volume is a compilation of 3512 corrosion and corrosion prevention articles, books, and brochures published in 1948 and 1949. Abstracts of 30 technical societies were canvassed regularly for material taken from more than 500 sources the world over. The NACE Abstract Filing Index, formulated by corrosion experts, is used to classify the material topically. Literature is placed in eight main groups: general; testing; characteristic corrosion phenomena; corrosive environment; preventive measures, materials of construction; equipment; and industries.

From Mine to Market, by Joseph T. Lambie. *New York University Press.* $6.00, 380 pp., 1954.—The Norfolk & Western R.R. is a uniquely specialized line whose prosperity depends on an immense volume of coal traffic. The book traces the growth of the road from its very beginnings. The closing chapter is an assessment of the probable future effects of the newer sources of energy. The entire book is carefully documented from the archives of the Norfolk & Western.

Proceedings, University Research Reactor Conference, edited by W. W. Grigorieff. *Oak Ridge Institute of Nuclear Studies,* $1.35, 221 pp., 1954—Information presented in formal papers, round table discussion, and in question periods at the University Research Reactor Conference is covered in the proceedings. The conference had three objectives: to summarize plans of educational institutions already owning, or planning to build reactors; to discuss specific problems of low power reactor construction, such as availability of fuel, personnel requirements, and cost; and to examine the educational significance of research reactors.

Symposium on Fluorescent X-Ray Spectrographic Analysis. *American Society for Testing Materials,* $1.75, 72 pp., 1954—The book is a compilation of papers presented at the 56th Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J., June 1953, in the symposium sponsored by the ASTM Committee E-2 on Emission Spectroscopy. Pictures, graphs, charts, tables, and schematic diagrams are used.

An important message concerning a new —and unique— alloy steel

UNITED STATES STEEL is pleased to announce to all steel consumers a new steel which we feel sure will enable you to improve the performance, lengthen the life, and reduce the cost of industrial equipment. This new engineering material is USS Carilloy T-1 steel, which recently underwent some very severe testing in Birmingham, Alabama.

The development of T-1 steel began back in 1947, when our research people took a good look at a problem that had plagued alloy plate steel users for years. That problem was the multiplicity of compositions required to do everyday jobs. There was no one composition available that could serve a wide variety of applications—so our research people set out to develop such a steel. The steel we wanted had to possess many unusual attributes such as very high strength, unusual toughness, good weldability and excellent resistance to impact abrasion at temperatures from minus 150°F. up to as high as 900°F.

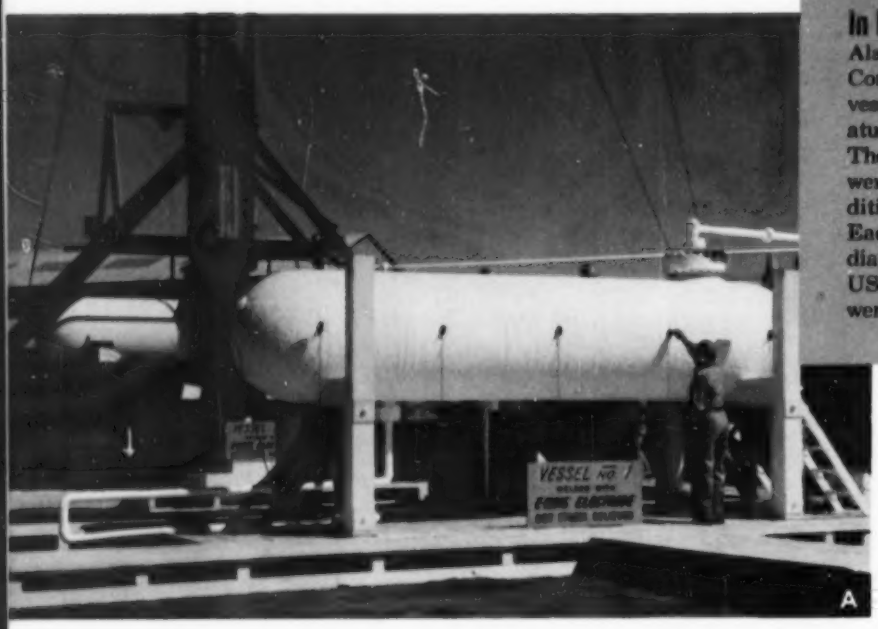
Today such a steel is a reality. It is USS Carilloy T-1 steel, which has been tested thoroughly, both in the laboratory and in actual service applications. To prove further the capabilities of this new Carilloy steel, Chicago Bridge & Iron Company and United States Steel this summer carried out a joint testing program on commercial pressure vessels built from T-1 steel. The results of these tests and their significance to all steel users are described on the next three pages. We feel sure that you will find the story well worth reading.

UNITED STATES STEEL

Remarkable new engineering material

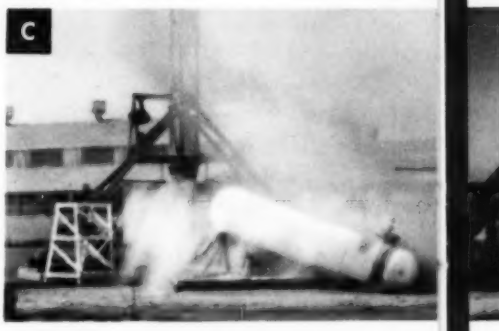
PROVES SUPER-TOUGH

In May and June, 1954, at the Birmingham, Alabama, plant of Chicago Bridge & Iron Company, eight welded cylindrical pressure vessels were refrigerated to subzero temperatures and deliberately tested to destruction. The vessels were all standard designs that were built under normal production-line conditions by Chicago Bridge & Iron Company. Each vessel was twenty feet long, four feet in diameter, and made of half-inch plates of USS Carilloy T-1 steel. Four of the vessels were welded without stress relieving, using



A

B


C

D

In pressure test at 38°F. below zero T-1 steel withstands stresses 3 times its design strength

This vessel, not stress relieved, was refrigerated to −45°F. Then, with a quarter inch of white frost clinging to its sides, a high pressure pump slowly chugged the pressure in the vessel up to 938 psi. At that point, the steel itself was stressed to 45,000 psi., which is one half the yield strength of T-1 steel.

The hydraulic pressure was forced still higher, to 1875 psi. At this pressure, the stress in the steel had reached 90,000 psi.—the full *nominal* yield strength. Still, every inch of the metal was sound and all welds were intact.

Now the test really came. The pump labored on, and the pressure climbed to 2,000 psi. . . . to 2500 . . . to 2850 psi. . . . then . . .

BOOM! A jet of yellow brine burst out of the vessel and shot 200 feet through the air. The vessel finally failed at a pressure of 2850 psi. and a minimum stress of 136,000 psi. on the plates. This stress is well over three times the design strength of 45,000 psi.

The temperature of the steel at the time of failure was a frigid 38 below, yet the metal showed no sign of brittle failure. Super-tough USS Carilloy T-1 did what no ordinary steel could do at such a low temperature—it stayed *tough and ductile;* in fact it actually stopped the tear from propagating any further through the vessel.

USS

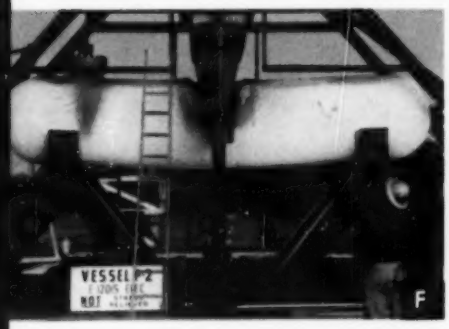
UNITED STATES STEEL

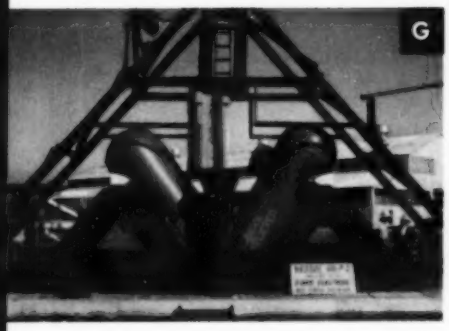
USS CARILLOY T-1 STEEL

DOWN TO 38°F. BELOW ZERO

E 12015 low hydrogen coated electrodes. The other four were welded with E 9015 electrodes and were stress relieved after welding. Two vessels of each type were given two tests: a pressure burst test for strength and ductility, and an impact punishment test for toughness and resistance to the most severe impact conditions that could be devised.

Results of these tests, described below, prove conclusively the exceptional strength and toughness of USS Carilloy T-1 steel, even at temperatures far below freezing.





Frozen steel vessel made of Carilloy T-1 survives blow from 13-ton ingot dropped 73 ft.

A steel ingot weighing 26,700 lbs. has just plummeted 52 feet and crunched on top of this test vessel (Fig. E). The ingot was traveling 39 miles per hour when it hit. It struck with an impact energy of about 1,400,000 ft. lbs., bounced fifteen feet, and crushed down again on top of the vessel.

This vessel was welded without stress relieving. It was refrigerated to 33°F. below zero. It was pressurized to 1,875 psi. equal to a stress on the plates of 90,000 psi.—the steel's *nominal* yield strength. Yet, it did not fail. The ingot left only a 7/16 inch dent in the top, with a slight bulge on either side of the dent.

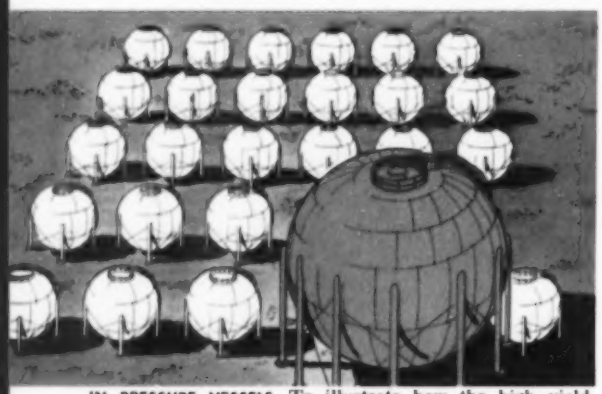
The test was repeated (Fig. F). This time the ingot was raised 73 feet, then dropped. It pounded down again on the very same spot on top of the vessel. Traveling at 46.7 miles per hour, it hit with an impact blow of 1,960,000 ft. lbs. The dent merely deepened – the steel and all welds were still intact.

Again the test was repeated (Fig. G). Now dropped from 101 feet, the 13-ton ingot ploughed into the top of the vessel at a speed of 55 miles per hour, hit with an impact of 2,750,000 ft. lbs. This time the vessel failed. But it didn't shatter. It didn't crack. It tore open like a tough piece of hickory. In other words, it failed without any signs of brittleness, even though the temperature of the steel was now minus 22°F.

In addition to this tremendous resistance to impact abuse, combined with remarkable sub-zero toughness, Carilloy T-1 steel also gives you excellent resistance to impact abrasion . . . good high temperature strength . . . and exceptional weldability.

Turn the page to see how you can use this remarkable new steel to cut costs and improve performance in a great variety of products.

Here's where you can reduce costs with USS Carilloy T-1 steel



IN PRESSURE VESSELS. To illustrate how the high yield strength of Carilloy T-1 steel can pay off here, consider this fact: To store 25,000 bbls. of propane at 210 psi would ordinarily require 25 mild carbon steel vessels. In contrast, only *one* large vessel made of T-1 steel of the *same* thickness would do the job. This single T-1 vessel would require 1/3 less shell material, 14% less foundation concrete, 70% less welding and 84% less space.

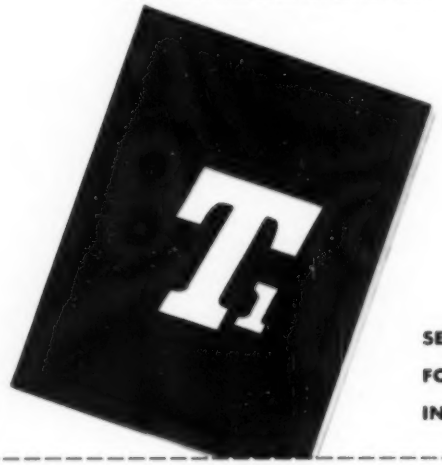
IN STEEL MILLS. In skip cars, T-1 steel in the bottoms, sides, and bail plates lasts three times longer than steels now in use. T-1 steel also gives exceptionally long service in coke bins and chutes, in ore transfer cars, in draft lines, in conveyor chains, and crane hooks. In clamshell buckets, T-1 steel has taken heavy abuse at temperatures from 500° to 600° F. and lasted *nine times* as long as the wear resisting steel previously used.

IN EXCAVATING EQUIPMENT. The dipper stick, bail, and bucket of this big electric shovel are fabricated of T-1 steel plate. The superior strength and durability of Carilloy T-1 made it possible to increase the capacity of the bucket from 35 to 45 cu. yds.

• The pressure vessel tests described on the previous pages indicate only one of the many possible uses of Carilloy T-1 steel. For this new engineering material has advantages not only in pressure vessels and storage tanks, but it also has proved itself in power shovels, bulldozers, mining machines, mine cars, steel mill ladles, blast furnace draft lines, lift trucks—in high speed rotating machinery, in stamping or forging presses. It is being considered for use in tension members of cantilever bridges and other similar applications where tension members are involved.

With the 90,000 psi. minimum yield strength to work with, combined with extraordinary sub-zero toughness, good high temperature strength to 900° F., and excellent resistance to impact abuse, impact abrasion, and atmospheric corrosion, you can use T-1 steel to reduce the size and weight of heavily stressed parts. This cuts your shipping and handling costs, as well as the cost of material, and the cost of foundations and supports where they are needed. Carilloy T-1 steel lengthens the life of your equipment, cuts repair bills and outage time. And remember, wherever you use T-1, you can weld or flame cut it either in the shop or in the field without expensive heat treating equipment. This cuts costs still further.

UNITED STATES STEEL CORPORATION, PITTSBURGH
COLUMBIA-GENEVA STEEL DIVISION, SAN FRANCISCO
TENNESSEE COAL & IRON DIVISION, FAIRFIELD, ALA.
UNITED STATES STEEL SUPPLY DIVISION, WAREHOUSE DISTRIBUTORS
UNITED STATES STEEL EXPORT COMPANY, NEW YORK



SEND COUPON FOR COMPLETE INFORMATION.

United States Steel, Room 4481
525 William Penn Place
Pittsburgh 30, Pa.

☐ Please send me your booklet "United States Steel presents T-1" which contains the full story of T-1 steel.

☐ Have your representative get in touch with me.

Name .....

Address .....

City ..... State .....

USS

UNITED STATES STEEL

. Industrial Notes . .

• *Photosensitive, anodized aluminum sheets for use with standard darkroom techniques, will be manufactured by Metalphoto Corp., under an arrangement with Horizons, Inc., giving the former company complete rights. Applications of the process are said to be especially efficacious where resistance to abrasion, temperatures up to 1000°F, and acids, salts, and organic solvents are important considerations.*

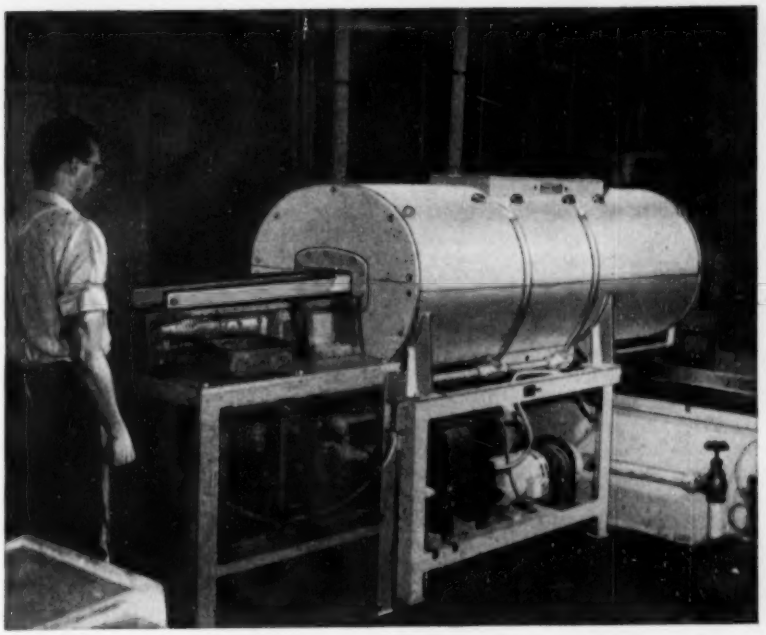
• **Salem-Brosius, Inc. and Phillips Corp., while completely maintaining separate corporate identities, have agreed to pool production facilities. Salem-Brosius will lease 45,000 sq ft of space in the Phillips factory building adjacent to the former's plant in suburban Carnegie, Pa. Salem-Brosius will use the space to carry on manufacturing activities for both companies.**

• World Steel Corp., Chicago, Ill., has applied to the Atomic Energy Commission for authority to use an atomic reactor in connection with making steel in a new plant to be constructed in the Chicago area. It is understood that World Steel plans to use the reactor for heating water into steam to operate turbines for electric power.

• The more theoretical aspects of Jones & Laughlin Steel Corp. research will be moved to the new center under construction at an initial cost of $1.5 million. The laboratory, to be completed in mid-1955, will be located at Baldwin Hill overlooking J&L's Pittsburgh Works. When the buildings are completed research now going on at existing locations will for the most part continue.

• **Lukens Steel Co. has increased heat treating facilities and is now heat treating, quenching, and drawing plates up to 60 ft long. In addition to carbon and low alloy steels, special alloys such as Iconel are being processed.**

• National Lead Co. announced selection of firms to perform construction on expansion of the nickel plant at Nicaro, Cuba. The construction companies are Frederick Snare Corp. and Merritt-Chapman & Scott Corp., both of New York City and Havana, Cuba. They will participate jointly in the project. National Lead made the selection as the operating contractor. General Services Administration, by directive from the Office of Defense Mobilization, is responsible for both operation and expansion of the plant. Office of Defense Mobilization authorized an investment up to $43 million for expansion of the plant by 75 pct.



IBM Improves Heat Treating Quality Cuts Costs With Hevi Duty Shaker Hearth Furnaces

At **IBM** — heat treated parts must meet rigid physical specifications and tolerances. Formerly a liquid cyaniding process was used and production was slow and costly. By using Hevi Duty Shaker Hearth Furnaces, the quality of the work has been improved and production has been increased.

The metallurgists at **IBM** state —

Controlled Case Depth — A uniform case depth of .002" to .015" is obtained. Three zones of temperature control assure an even temperature for the entire length of the hearth plate. The flexibility of dry cyaniding enables our operator to produce a case which is most suitable to the type of steel, shape, and function of the particular part.

Uniform Hardness — is obtained because all parts fall from the hearth into the quench individually. This is especially true of small studs which were formerly dumped from baskets into the quench.

Less Distortion — because in these furnaces the parts are not pushed or packed together. The work is heated and quenched at a rate which minimizes internal stresses and possible warpage. The warpage is less than that experienced with careful handling by former methods.

Clean Bright Surface — is a requirement because the work is flash chromium plated. Washing and cleaning time has been reduced.

The Hevi Duty Shaker Hearth Furnace is fully adjustable to give complete control of heat treating processes such as Hardening, Dry Cyaniding, and Carburizing. Write for Bulletin HD-850 which describes the furnace that assures uniform results on a production basis.

HEVI DUTY ELECTRIC COMPANY

HEAT TREATING FURNACES HEVI-DUTY ELECTRIC EXCLUSIVELY

DRY TYPE TRANSFORMERS — CONSTANT CURRENT REGULATORS

MILWAUKEE 1, WISCONSIN

THAT willfull child of the 20th century, atomic power, is beginning to show signs of growing up. Whether it will ever reach an age where it's more rambunctious tendencies are curbed depends largely on environmental factors—which the Soviets play no little part in creating. Three news events, almost tumbling over each other in their sudden appearence upon the American scene, bode well for the future. The first was the passing and eventual signing of the new atomic energy legislation fought for by the President.

When President Eisenhower signed the new law he opened the way for a future private atomic industry. Passed after much debate, vituperation, and adherence to party lines, the law also provides for international cooperation between the U. S. and allied nations. Under certain security regulations the U. S. can now share atomic knowledge with friendly countries.

While the bill has become law there are several provisions that the 84th Congress is expected to reinvestigate. One matter is the compromise reached after 13 days of filibuster which requires that companies share for five years any atomic power patents.

The law allows the Atomic Energy Commission to grant 40-year renewable licenses for private industries to operate atomic facilities and to handle fissionable materials. The Government retains title to all fissionable materials discoveries. Publically-owned utilities and cooperatives are to receive first preference for use of publically-owned power developed by AEC experimental plants.

The U. S. can now share secret details on the external characteristics of atomic weapons and also is in a position to supply allied nations with methods of performance and defenses against atomic war.

The second and third events came simultaneously. President Eisenhower waved a neutron wand to start excavation for the first commercial electric plant in the nation to be powered by atomic energy. The President, in Denver, Colo., activated an unmanned 14-ton shovel some 1200 miles away in Shippingport, Pa., which scooped up three tons of earth, moved forward, and then dumped the load. The plant is a joint project of the AEC, Duquesne Light Co., and Westinghouse Electric Corp. Rear Admiral E. G. Rickover, the commission's director of the project, said that Government's share would be about $33 million, with Duquesne paying about $13 million toward the plant cost. While the cost of producing electricity using atomic power is expected to be greater than by conventional coal burning plants, designers hope to eventually produce an economically competitive atomic power plant.

Westinghouse is building the reactor under contract with the AEC. Duquesne will build the generating part of the plant at its own expense. It will operate the plant and pay the commission for steam generated by the reactor—whose ownership is to be retained by the Government.

Third on the list of events was the announcement from the President that a world agency for the dissemination of atomic energy knowledge is being formed. Prospective members of the pool are Great Britain, Canada, Australia, South Africa, and France. There will be others. Talks are in progress with several nations. Negotiations are also underway for the construction of an atomic power reactor in Belgium. Raw uranium and fissionable materials will be set aside for agency use, the President announced. Disclosure of the move toward formation of the pool came during ceremonies connected with the Shippingport project.

President Eisenhower first broached the world atomic pool idea before the United Nations last year. What part the UN will play in the agency is yet undetermined. Thus far, the attitude of Russia toward the pool has been almost completely negative. One source pointed out, however, that political embarrassment finally forced the USSR into the Technical Assistance program after many years of noncontribution. In his UN speech, President Eisenhower said that the agency would aim at the application of atomic energy to medicine, agriculture, and other peaceful activities. Speaking to the audience watching the Shippingport ceremony, President Eisenhower said: "We have just agreed with a number of other nations to go ahead now with the formation of an international agency which will foster the growth and spread of new atomic technology for peaceful use. Atomic materials for projects sponsored by this agency will be set aside for that purpose. We hope that no nation will long stand aloof from the work of this agency."

Participation in the agency by the U. S. is subject to Congressional approval under amendments adopted in the new atomic energy law. Press Secretary James C. Hagerty noted that the project had nothing to do with weapons. There was no immediate reaction from Moscow. Tass was slow in reporting the move in the Soviet press and major government figures were reported to be out of town on that particular weekend.

◆◆◆◆◆◆

WHEN Congress closed shop last summer it left the fate of the Texas City tin smelter hanging fire. The Senate failed to get Representative Jess Wolcott's resolution out of committee in time for a vote. Under the terms of the bill, previously passed by the House, an equal number of representatives and senators were to delve into the necessity of the tin smelter. Life of the installation has been extended to June 30, 1955, pending a report on its usefulness.

The study must be completed and the report presented by Mar. 15, 1955, in accordance with the measure extending operation of the unit. Now, it will be up to the 84th Congress to do the work. Opponents of the smelter claim it is producing tin not needed by the nation, for industry or stockpiling.

Originally, Senator Wolcott planned to investigate the nation's foreign sources of tin along with the smelter. It is his opinion that if the U. S. must depend upon overseas areas for the metal that at least

it should have some idea of the factors bearing on its availability.

Representative Wolcott maintains that "If we must rely on these distant sources, it would seem the part of wisdom to have first-hand knowledge of the conditions on which these sources depend for their existence."

Present smelter policy requires that all tin produced at Texas City go into the national stockpile. None of it is available for civilian use.

◆◆◆◆◆◆

ALMOST no one would deny that the Russian steel industry has made tremendous strides since the end of World War II. After the Germans were driven out many USSR metallurgical facilities had to be rebuilt from the ground up. By 1953, the Soviets were producing 41.8 million short tons of steel annually—exhibit A in the communist showcase. However, items appearing in Pravda, official party organ, give one the feeling that all is not sweetness and light. Lately, the party newspaper has been chastising the Russian worker for certain evident failings.

For one thing, the Russian worker has been resisting modernization. He sees machines as huge scissors cutting into his paycheck. The truth seems to be that the Soviet laborer would rather work long hours for more pay than short hours, more production—and less cash.

An editorial in the August 10 issue of Pravda stated that "It is imperative to step up the pace of increasing capacities of ferrous metallurgy and expanding the metallurgical base of the country." Last year, Pravda scolds, several blast furnaces, open hearths, rolling mills, coke batteries, and concentration and sintering plants failed to go into operation. The program this year is not up to snuff, either, according to the communist editorialists.

Existing plants are not meeting planned production rates, according to Pravda, because of unwillingness to put technological advances to good use. New machines lay idle while manual labor continues to do the job. Labor productivity in one of Russia's pet industrial areas, the Ukraine, was reported only 90 pct of plan in the Kirov Frunza plants. The gripe is that the plants are not using all their reserves. It was *suggested* that unused time be employed to increase production of metals at various plants.

"Intolerable waste is permitted which reaches up to 2.5 pct of total steel output," Pravda charges. The newspaper also asserts that many plants were not attempting to lower production costs, improve quality, and produce scarce rolled metal.

Trouble seems to be rampant in the Crimea, too. Complaints there seem to be concerned with the fact that "plants and mines are not fulfilling state plans or using resources at their disposal." Among other things scrap flow in the Crimea appears to be less than good. Scrap collection was described by Soviet news sources as badly organized at Kerch, Yalta, Sevastopol, and Fedoosia.

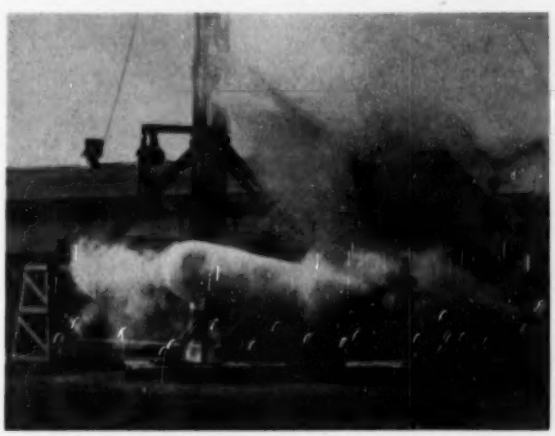
With all the troubles so loudly declared by Pravda, some experts on this side of the Iron Curtain estimate that Russian steel production may reach 45.5 million short tons for 1954.

◆◆◆◆◆◆

OPERATION T-1 can at least be described as spectacular. Under a blazing hot summer sun in Birmingham, Ala., U. S. Steel Corp. put a new kind of steel through some rugged burst and impact tests to prove to government and technical code authorities, Atomic Energy Commission representatives, Army and Navy officers, and military and civilian aeronautical groups that Carilloy T-1 can take it. U. S. Steel, through the medium of the tests, attempted to prove: the fitness of quenched and tempered plate for the construction of pressure vessels; the propriety of utilizing the great strength potential of T-1; the desirability of designing on the basis of yield strength rather than ultimate strength; and the suitability of modifying the present requirements for stress relieving so far as they pertain to toughness.

Before the tests began vessels made of the new quenched and tempered steel were coated with ice at −45°F. A new method for testing full scale vessels was used in the drop tests. A 26,700 lb steel weight was allowed to fall on the tanks from 52, 73, and 101 ft to show the toughness of the steel at low temperatures. Internal pressure during the drop tests was 1875 lb per sq in. At the start of the tests the four tanks used appeared identical—20 ft long, including hemispherical ends, and 48 in. in diam, made from ½ in. thick plates, and crusted with white frost. However, the two tanks tested on the first day were not stress relieved, but rather left in the natural condition as welded. The second set, used the next day, were stress relieved.

Both the non-stress relieved vessel and the stress relieved unit burst under internal pressure of 2850 lb per sq in. In the drop tests the two types burst from the same force—the 26,700 lb weight dropped from a height of 101 ft.



Icy calcium chloride geysers out of steel tank frozen at −45°F. Tank burst from internal pressure of 2850 psi. U. S. Steel Corp. developed the new type steel, T-1.

for copper converter linings . . .

These carefully controlled refractories are made from the best quality raw materials, are formed under high pressure and are fired extremely hard. Dependable workmanship and uniformity are assured.

In addition, Harbison-Walker produces various other classes of basic refractories for every metallurgical furnace need.

HARBISON-WALKER

provides Two service-proved basic refractories

• Chromex B

Briefly summarized, the desirable properties which account for the excellent service secured with this extra hard fired chrome-magnesite refractory are:—high density and impermeability—relatively low thermal expansion and good resistance to thermal shock—unusual stability and high strength at high temperatures.

• H-W Magnesite

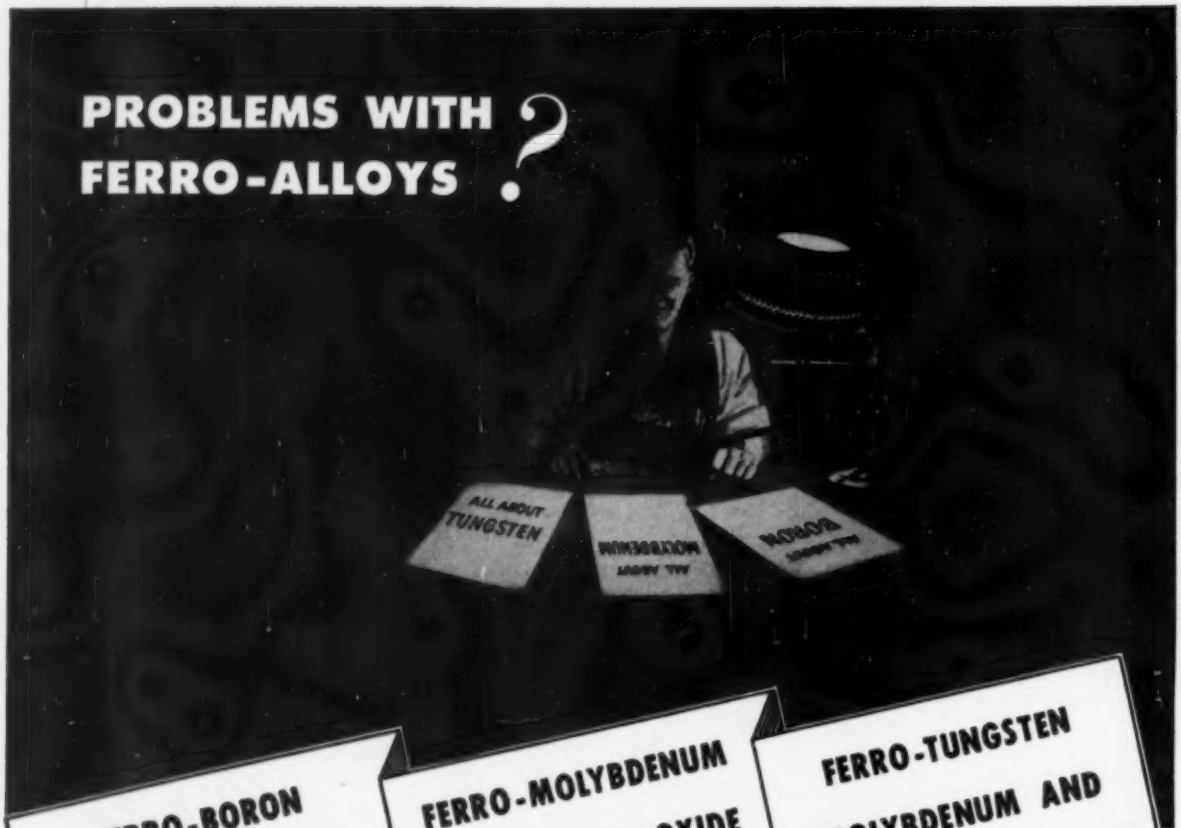
For the most strongly basic linings this refractory with the magnesia content of over 90% fulfills the most severe requirements. It is fired at an extremely high temperature and is dense, strong and volume stable at temperatures far above the highest employed in copper converters.

HARBISON-WALKER REFRACTORIES COMPANY

AND SUBSIDIARIES

General Offices: Pittsburgh 22, Pennsylvania

World's Largest Producer of Refractories



FERRO-BORON
BORON DEOXIDIZERS
BORIDE DEOXIDIZERS

FERRO-MOLYBDENUM
MOLYBDENUM OXIDE
RARE EARTHS

FERRO-TUNGSTEN
MOLYBDENUM AND
TUNGSTEN METAL POWDERS

CHEMICALS OF MOLYBDENUM...TUNGSTEN...BORON

Molybdenum is now available in unrestricted supply to improve strength and machinability. Dependable results are still one of its major attributes.

Tungsten, for hardenability and wearability improvement is now used in surprisingly small additions, with great success.

Boron, as an intensifier of the effects of other alloying materials, may be used in very minute additions, and yet maintain the essential properties of the castings desired. The most economical and satisfactory form to introduce Boron is recognized to be found in MCA's Ferro-Boron.

Operating the world's largest rare earth deposits, the Molybdenum Corporation of America has recently conducted extensive pioneering research in evaluating the properties, applications and uses of RareMeT Compound.

In nodular iron, small additions of rare earths have helped to produce consistently good ductility by counteracting subversive elements such as lead and titanium.

Write today for further information.

MOLYBDENUM

Grant Building CORPORATION OF AMERICA **Pittsburgh 19, Pa.**

Offices: Pittsburgh, Chicago, Detroit, Los Angeles, New York, San Francisco
Sales Representatives: Edgar L. Fink, Detroit; Brumley-Donaldson Co., Los Angeles, San Francisco
Subsidiary: Cleveland Tungsten, Inc., Cleveland Plants: Washington, Pa., York, Pa.

MCA

Drift of Things

WITH the launching of the Metals Branch Research Publication Fund described on p. 1165, the Metals Branch plans to balance its budget and still continue publishing the increasing number of Metals Transactions papers. President Reinartz was in New York the other day, and we got into a conversation with him on the general subject of Transactions.

"Transactions," said Mr. Reinartz, "are the records of the pioneers in the mining, metallurgical, and petroleum professions. The theories and principles which these men evolve, later become a part of normal engineering practice.

"The AIME Transactions are the vital media for the presentation and exchange of ideas. They have become, because of their unimpeachable integrity, the life blood of the profession and of the AIME."

After our conversation we looked up some records on the subject. Taking the record of last year as an example, 197 papers were submitted for Metals Transactions. Of these papers 23 pct were declined, 24 pct accepted after major revision, 39 pct accepted after moderate revision, and 14 pct were accepted as submitted. This represents a tremendous investment of time and effort. Each one of the submitted papers is carefully checked by not just one but by at least *three* recognized authorities on the subject. These men give unselfishly of their time to insure that papers published by AIME are a genuine creative contribution to the profession.

The work of these experts and of the men who make up the Technical Publications Committee is usually unheralded or unknown. But these are the men to whom the Institute points with pride as responsible for upholding the unimpeachable integrity of Transactions to which President Reinartz referred.

The system of checks and balances which the Institute has evolved over its years of experience assures the reader that in AIME Transactions he has the science of his profession at its finest. By encouraging high standards of work, the Institute places the best available and most timely data at the constant disposal of the engineer. It seems to us that this is the combination which makes for success and progress in our profession. (E.M.W.)

QUITE often one finds it difficult to put thoughts into words, and then unexpectedly comes across the words of another expressing the same ideas. In this case, we found our thoughts spelled out in the September issue of *Industrial & Engineering Chemistry*, one of the publications of the American Chemical Society. As noted in the first item on this page, AIME Transactions represent the fundamental tools of our profession. With the maintenance of high standards as one of our basic responsibilities, we quote an editorial written by Walter J. Murphy, Editor of ACS publications, who has obviously given much thought to a general problem.

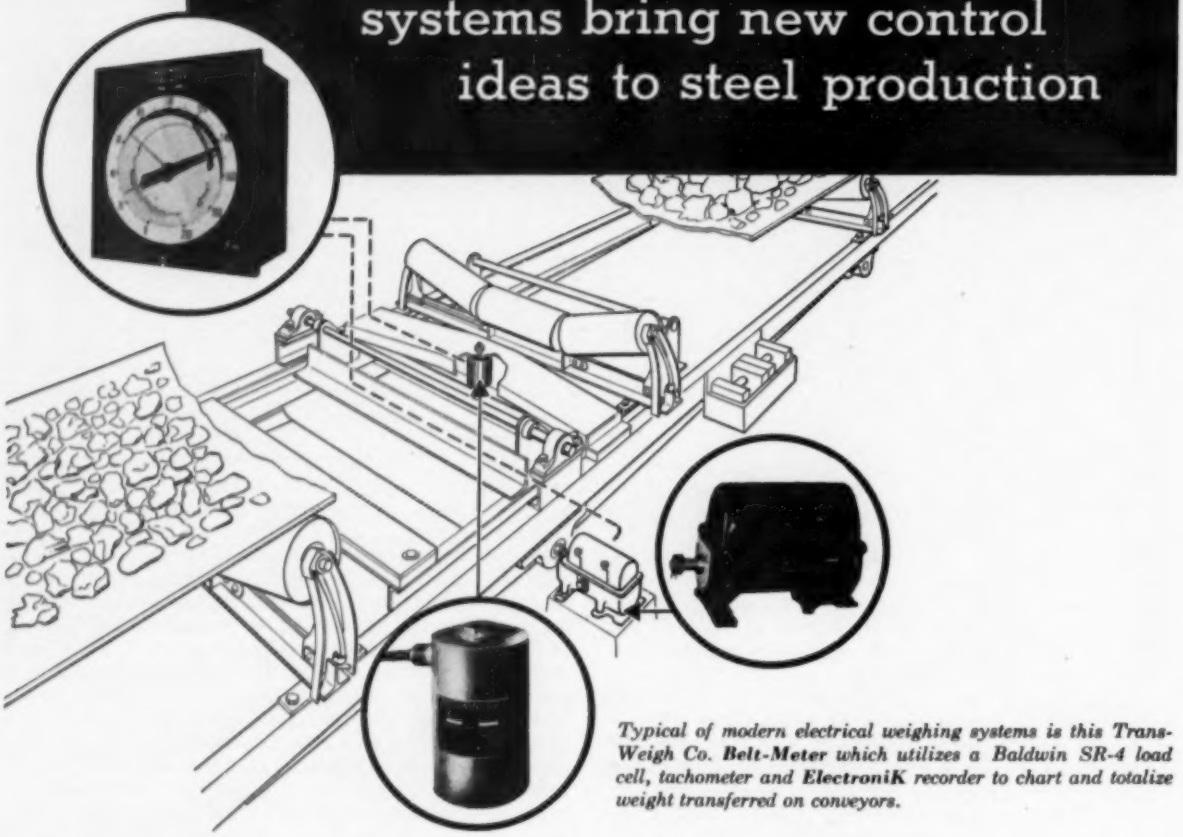
"Many old sundials carry the Latin inscription 'Non nisi numero horas serenas'—'I count only the happy hours.' We wish the same sort of scale could be applied to evaluation of the worth of scientists, particularly professors and graduate students. Through the years an insidious form of merit determination has been woven about us all. It is the judging of a scientist's merit by papers and patents —the sheer physical number—for which he has credit. We have been aware of this in a general way; so have thousands of others. But we were set back on our feet and rocked a short time ago by a plain statement from an author to a proposal by us. To save space, we suggested combining three of his papers, related in subject matter, under a single title and authorship. This is what he said: 'We are anxious to have *three* papers, not *one*. We would have obtained this situation quite readily if we had submitted each of them separately. It appeared to us, however, that it would be proper to have the papers appear together. Unfortunately, the fact remains and no doubt will remain for a long time in job applications for our graduate and postgraduate men, that the number of papers a man has is important.' We do not recall that any author has been quite so blunt before. We do not blame this professor particularly—he is caught in the same web as hundreds of others. But we do find fault with the system that has led to the establishment of such artificial standards.

"It is true that research is part of the lifeblood of good teaching. Results of research which are significant should be published, for otherwise they are of value only to the men doing the research and others with whom they have personal contact. The ACS Committee on Professional Training, in evaluating chemistry departments for the Society's list of approved schools, takes into account faculty members' research activity and productivity. But (to quote from one of the published statements of the committee), 'The Committee . . . does not wish to encourage publication of short papers rather than substantial contributions, or to place emphasis on numbers of publications rather than their scientific value. It is generally recognized by all experienced chemists that one significant and outstanding paper may contribute more to the science than a large number of less fundamental or important papers.' Five articles on various phases of a continuing piece of work may add up to an impressive total of pages. But each must repeat to some degree material presented before. In the long run, perhaps two articles, at the end of the work, would have done the job adequately and made reference easier. We do not mean to imply at all that publication should be held up until the bitter end of a research project. This may lead to the other extreme—failure to report at all, leaving the research of little value except through the education of the men who did it or through transmission of the information by other means than publication. Publication of some of the results may save the repeating of some of the work or may lead to valuable suggestions. We *do* deplore the piecemeal papers which accomplish nothing more than to raise the total of an individual's published work."

In AIME, we have an outlet for publishing short and important research data—Technical Notes. More use should be made of this medium.

A. S. Cohan

Automatic electric weighing systems bring new control ideas to steel production



Typical of modern electrical weighing systems is this Trans-Weigh Co. ***Belt-Meter*** *which utilizes a Baldwin SR-4 load cell, tachometer and* ***ElectroniK*** *recorder to chart and totalize weight transferred on conveyors.*

MODERN methods of continuous electrical weighing of solids are opening up new ideas of process control in the steel industry. Utilizing Baldwin-Lima-Hamilton SR-4 load cells, *ElectroniK* recorders and Brown servo components, these systems offer distinct advantages.

The technique permits remote measurements, and is applicable to material on belts, conveyors, cranes, and in railroad cars, hoppers or ladles. Automatic control is often possible. The systems are rugged . . . can take appreciable overloads.

Among the leading equipment of this type now available are the following:

Trans-Weigh Co. **Belt-Meter** . . . (illustrated above) for weighing and totalizing material in transit. See Data Sheet No. 10.18-3.

Ametron Electronic Scale, made by Streeter-Amet Co., weighs material on crane hooks, track scales, etc. See Data Sheet No. 10.18-2.

Gilmore Electronic Scale, using Brown servo amplifier, is applicable to many weighing problems. See Data Sheet No. 10.18-4.

Brown Motion Transmitter works with existing mechanical scale to record weight, through linkage to the scale beam. See Data Sheet No. 11.5-2.

Your nearby Honeywell sales engineer will be glad to discuss your own applications . . . or, if you prefer, a representative of the manufacturer of the specific system will consult with you.

MINNEAPOLIS-HONEYWELL REGULATOR CO., *Industrial Division*, Wayne and Windrim Avenues, Philadelphia 44, Pa.

• **REFERENCE DATA:** Write for Data Sheets on specific weighing systems, and for Data Sheet No. 10.18-1a, "Baldwin SR-4 Devices."

MINNEAPOLIS **Honeywell**

BROWN INSTRUMENTS

First in Controls

ELECTROMET

Trade-Mark

ALLOYS

"Quality Plus Service"

Boron . . Ferroboron (in minimum 10.00% and 17.50% Boron grades), Manganese-Boron, Nickel-Boron and Boron Carbide.

Calcium . . Calcium-Silicon, Calcium-Manganese-Silicon, and Calcium Metal.

Chromium . . "Simplex" Low-Carbon Ferrochrome (0.010% and 0.025% maximum Carbon), Low-Carbon Ferrochrome (from 0.02% to 2.00% maximum Carbon), Nitrogen-Bearing Low-Carbon Ferrochrome, High-Carbon Ferrochrome, Exothermic Ferrochrome, Exothermic Silicon - Chrome, "EM" Ferrochrome - Silicon, "EM" Ferrosilicon-Chrome, "SM" Ferrochrome, Foundry Ferrochrome, Electrolytic Chromium Metal, and other Chromium Alloys.

Columbium . . Ferrocolumbium and Ferrotantalum-Columbium.

Manganese . . Standard Ferromanganese, Low- and Medium-Carbon Ferromanganese, Low-Iron Ferromanganese, "Mansiloy" Alloy, Manganese Metal, and other Manganese Alloys.

Silicomanganese . . In maximum 1.50%, 2.00%, and 3.00% carbon grades.

Silicon . . Ferrosilicon in all grades, including both regular and low-aluminum material, Silicon Metal, "SMZ" Alloy, Magnesium Ferrosilicon, and other Silicon Alloys.

Titanium . . Low-Carbon Ferrotitanium, Silicon-Titanium, and Manganese-Nickel-Titanium.

Tungsten . . Ferrotungsten, Tungsten Powder, Calcium Tungstate, and Calcium Tungstate Nuggets.

Vanadium . . Ferrovanadium in all grades, Vanadium Oxide (Fused), Sodium Polyvanadate (Red Cake), and Ammonium Metavanadate.

Zirconium . . 12 to 15% and 35 to 40% Zirconium Alloys, and Nickel-Zirconium.

"EM" Briquets . . Silicon, Silicomanganese, Ferromanganese, Chromium, and Zirconium Briquets.

Information about these and other alloys and metals produced by ELECTROMET is contained in our new catalog "ELECTROMET Ferro-Alloys and Metals." Write for a copy.

Electro Metallurgical Company

A Division of Union Carbide and Carbon Corporation

30 East 42nd Street UCC New York 17, N. Y.

OFFICES:
Birmingham • Chicago • Cleveland Detroit • Houston • Los Angeles • New York • Pittsburgh • San Francisco

WAREHOUSES:
Chicago • Los Angeles • Oakland, Calif. • Portland, Ore. • Sheffield, Ala.

IN CANADA:
Electro Metallurgical Company, Division of Union Carbide Canada Limited, Welland, Ontario

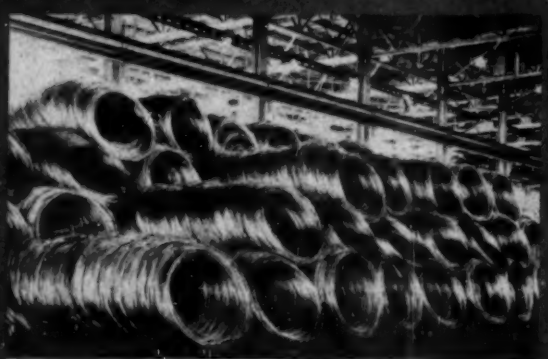
"Electromet," "EM," "Mansiloy," "Simplex," "SM," and "SMZ," are trade-marks of Union Carbide and Carbon Corporation.

Products of Sixty-Six Years of Pioneering in Aluminum

Your Alcoa Distributor is as near as your telephone, and his warehouse is stocked with the aluminum products you need for fast, economical production. His facilities include modern equipment for sawing, shearing and slitting stock to your specifications and for making prompt deliveries.

EXTRUDED SHAPES: For thresholds; window sills; door frames; glass stops; copings; gravel stops; trim; truck bodies. Also angles; channels; tees; zees; etc., for structures.

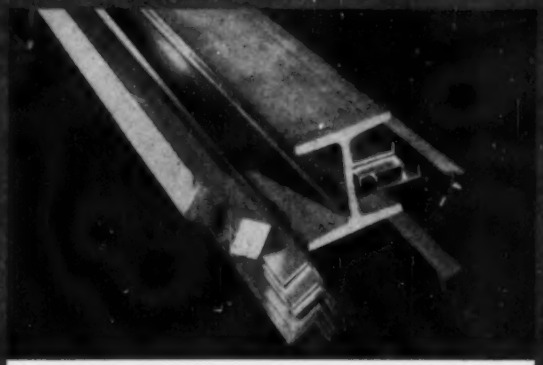
TUBE AND PIPE: Coiled tube; Alcoa® Utilitube; straight tube in round, square and rectangular shapes; heat exchanger tubes; standard pipe and pipe fittings; construction pipe; rigid conduit.



WIRE: Coiled and straight length; flattened wire; rivet wire and rod for die heading operations in manufacture of rivets, nails, bolts and screws.

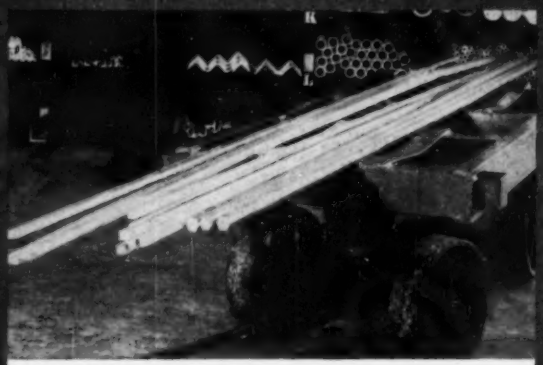
ALCOA ALUMINUM

ALUMINUM COMPANY OF AMERICA

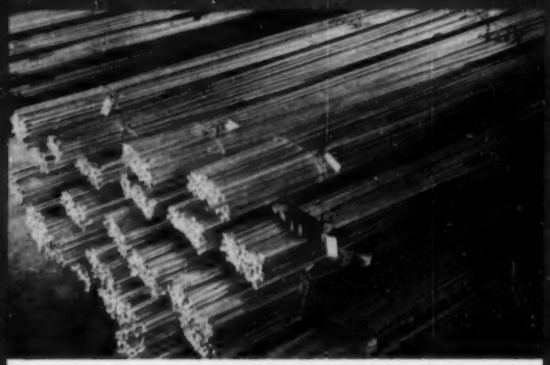


ROLLED SHAPES: Equal angles; unequal angles; channels; I-beams; tees; zees. Suited to a variety of structural applications.

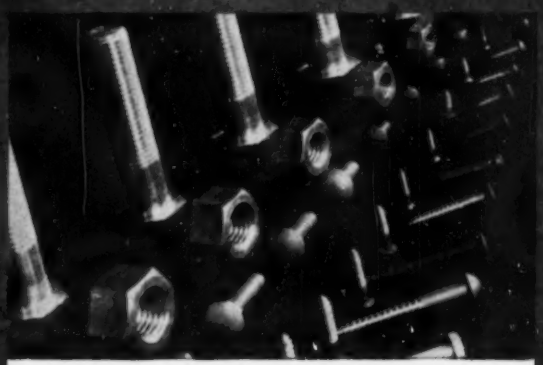
SHEET AND PLATE: Flat and coiled sheet; circles; patterned sheet; plate; tread plate; roofing and siding sheet; roofing accessories and fasteners; specialty sheet.



BAR STOCK: Square, hexagonal and rectangular in all commercial alloys. Rolled and cold finished to final dimensions for superior tolerance and finish.



SCREW MACHINE STOCK: Available in 2011-T3 (formerly 11S-T3), the free-machining alloy, and 2017-T4 (formerly 17S-T4), a higher strength alloy, plus 2024, 6061 and 7075 (formerly 24S, 61S and 75S).



FASTENERS: Machine screws, wood screws; washers; nuts; bolts; rivets. Recommended for fastening aluminum to prevent electrolytic damage.

WELDING AND SOLDERING: Welding and brazing wire; welding and brazing flux; solder flux; solder.

Conquers Heat, Erosion and Corrosion. Stainless steels containing nickel lengthen life expectancy of various components used in severe service. For instance, Diesel engine turbocharger rotors. Some rotors operate at from 12,500 to 21,000 r.p.m., and at temperatures upward of 1000°F. The rotor comprises a slotted disc of chromium-nickel stainless steel, with inserted blades of the same material . . . to take the brunt of attack in converting heat into mechanical power. Type "19-9" stainless, containing molybdenum and tungsten additions, is used to make sure of high tensile strength and low creep at the high temperatures encountered. And a somewhat similar austenitic chromium-nickel stainless steel . . . 18-8-S-Mo . . . is used for turbine nozzle blades.

STAINLESS PROVIDES THE ANSWERS . . .

Thousands of designers and engineers have found austenitic chromium-nickel stainless steels to be the key to new high standards of product performance and minimum operating costs. You, too, may find that a stainless steel containing nickel will help solve *your* metal problem.

1. At Elevated Temperatures

Stainless steels of this type possess the two essential qualities for successful use at elevated temperatures. By virtue of the chromium content, and assisted by the nickel content, they have outstanding resistance to scaling and oxidation. The nickel content also results in high creep strength and stress-to-rupture properties. Combined, these properties permit use of chromium-nickel stainless even in thin sections required for lightweight design.

2. At Sub-Zero Temperatures

Experience has shown that unalloyed steels become embrittled rapidly as temperature is lowered below normal. Nickel is by far the best alloying element to prevent brittleness. At sub-zero temperatures, chromium-nickel stainless steels provide unsurpassed impact values. These steels retain such a high level of toughness that they are known to be useful at temperatures as low as —400°F. and possibly even lower.

3. Under Corrosive Conditions

Selection of exactly the right alloy for your specific needs may be made from some thirty alloy steels qualified to carry the family name "Stainless." By virtue of their strength and resistance to attack by chemicals and atmospheres over a wide range of concentrations and temperatures, these austenitic chromium-nickel steels assure not only longer life of the equipment and purity of product, but also permit decrease of bulk and deadweight without sacrificing safety.

4. Where Erosion Is A Problem

Chromium-nickel stainless offers the utmost in resistance to erosion and cavitation. By cold working, it is possible to increase the tensile strength to as much as 300,000 psi. When subjected to erosion, the erosive media work hardens the surface and, correspondingly, curbs the attack. This resistance to erosion combined with corrosion resistance results in prolonged life for equipment made from the chromium-nickel stainless steels.

5. Use *Stainless* To Meet Complex Requirements

Utilize the unique combination of properties provided by chromium-nickel stainless steels when designing new products or improving old ones. You can draw, spin, forge, weld, solder, punch, shear or bend chromium-nickel stainless steels. Leading steel companies produce these austenitic alloys in all commercial forms. A list of sources of supply will be furnished on request.

EMBLEM OF INCO SERVICE

THE INTERNATIONAL NICKEL COMPANY, INC. 67 WALL STREET NEW YORK 5, N. Y.

Avery Adams, president of Pittsburg Steel Co., predicts that 1955 will be a better steel production year than this one. He envisions the upswing starting this October. Big gains this month will come from Detroit where new model production starts to roll in earnest.

Electro Metallurgical Co., plans to build a titanium metal plant near Ashtabula, Ohio to produce titanium by a method other than the Kroll process. The Electromet process involves sodium reduction of titanium tetrachloride. Electromet, a division of Union Carbide & Carbon Corp., plans to spend about $31.5 million for the new facility which will have an annual production of at least 7500 tons. The new process has been tried in a pilot plant at Niagara Falls, N. Y.

Britain and Russia are competing for contracts to build steel plants under India's second five year economic plan. At least one plant, and possibly two, are scheduled under the plan.

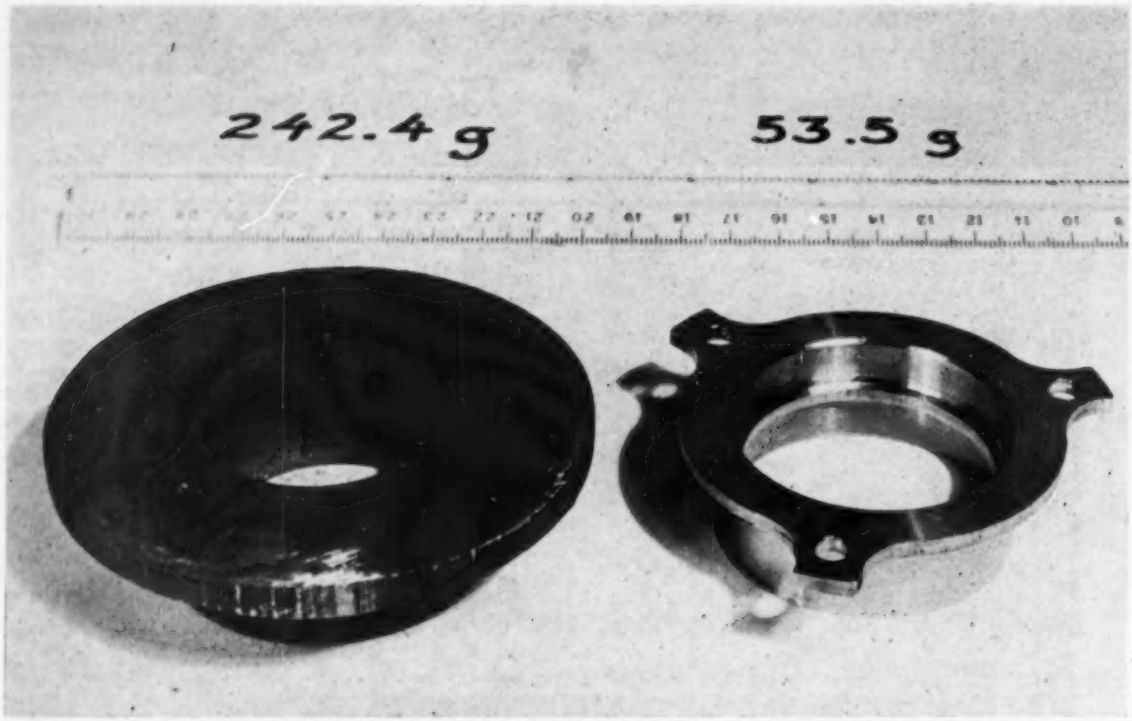
Reserve Mining Co., awarded the contract for construction of its new E. W. Davis Works at Silver Bay, Minn., to Arthur G. McKee & Co. The taconite plant is the result of extensive pilot plant research on the part of the company. Cost of the installation will be $19 million.

Republic Steel Corp.'s desire to move equipment purchased from Follansbee Steel Corp., to Gadsden, Ala., had civic leaders of Follansbee, W. Va., up in arms. Included in the deal are two cold reducing strip mills, one cold finishing strip mill, two continuous cold sheet mills, auxiliary equipment, and two tinning pots for long terne plate. Residents charged that the move would turn Follansbee into a ghost town.

Security regulations have been thrown up around a "new and improved" $2.5 million uranium refining process for the Port Hope, Ont., uranium refinery of Eldorado Mining & Refining Ltd., owned by the Canadian Government. Catalytic Construction of Canada Ltd., wholly owned subsidiary of Catalytic Construction Co., Philadelphia, is engineer and builder for the project. Completion is set for 1955.

World bauxite production in 1953 reached an all-time high at 13.8 million long tons. U. S. output was 5 pct lower than in 1952, according to official figures of the Bureau of Mines. The report was prepared by George C. Branner under supervision of F. J. Cservenyak, chief, light metals branch, minerals div. Imports were 2.8 times domestic output last year indicating an increased dependence on imports, a trend which started in 1947.

It has been reported that the Justice Dept., may attempt to prevent the merger of Bethlehem Steel Corp. and Youngstown Sheet & Tube Co. Washington sources say that the Justice Dept. has decided to withhold assurances that no trust-busting law suit will be instituted.



Comparison of hot press blank and finished machine part illustrates the degree of utilization of starting material. Parts weighing up to 125 lb and as large as 9 in. in diam have been produced.

Hot Pressing, Press Forming Loom As Answers to Titanium Fabrication

by H. W. Dodds and G. F. Davies

BRUSH Laboratories has developed two powder metallurgy methods for titanium fabrication which meet the rather stringent requirements for handling the metal. Conventional methods for fabricating titanium to finished tolerances are casting, die casting, forging and other processes. Brush's methods, it is felt, satisfy primary requisites of uniform quality of the end product both chemically and metallurgically. Oxygen, nitrogen, carbon, and hydrogen contamination are restricted to a minimum level and no greater than that of the starting material, titanium sponge.

Hot pressing and press forming are the answers offered by Brush. Developing the processes required a means for powdering titanium which kept gaseous and metallic impurities as low as possible. This kind of product is now produced by comminuting the commercial grade of ductile titanium sponge.

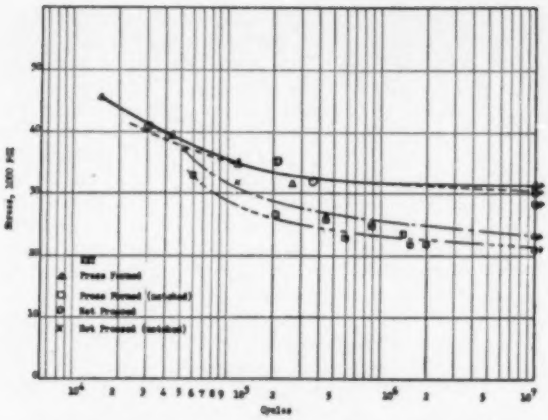
Hot Pressing

In the hot pressing method, high vacuums and temperatures are used to prevent gaseous contamination and to remove volatile constituents such as magnesium and magnesium-chloride. Graphite dies have been found satisfactory for producing high density, ductile parts having dimensional tolerances of approximately 1/16 in. depending entirely upon the shape of the part. In some cases, closer tolerances can be achieved. Parts weighing as much as 125 lb and as large as 9 in. diam have been produced with present equipment. Fabrication by this method lends itself readily to prototype production of contoured cored shapes normally produced by casting methods. The utilization factor on intricate parts is generally less than 4.5 lb of sponge to make a 1 lb finished machined part. Surface contamination by the graphite is kept at a minimum; and for most parts, the contamination is less than 0.005 in. deep.

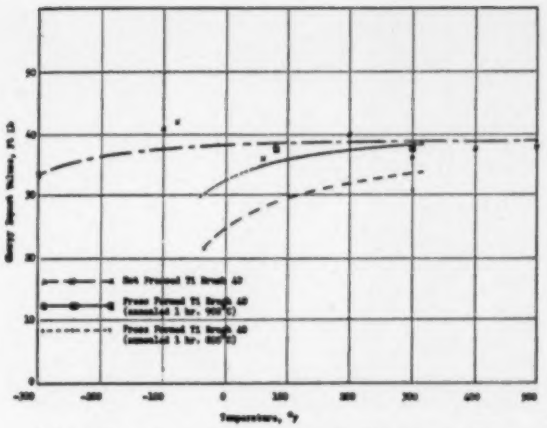
Press Forming

The press forming method is ideally suited for the production of large quantity, low cost parts. Blanks or semifinished shapes are pressed from powder and sintered in vacuum; and as in hot pressin, this processing tends to remove volatile constituents. The pieces are then given a series of forming operations to produce a part of finished shape. Operations such as forming, extruding, rolling, and draw-

H. W. DODDS and G. F. DAVIES are Vice President In Charge Engineering Services and Staff Engineer respectively, Metallurgical Dept., The Brush Laboratories Co., Cleveland.



Notched and unnotched fatigue strengths of Ti-Brush 40 produced by hot pressing and press forming as shown.



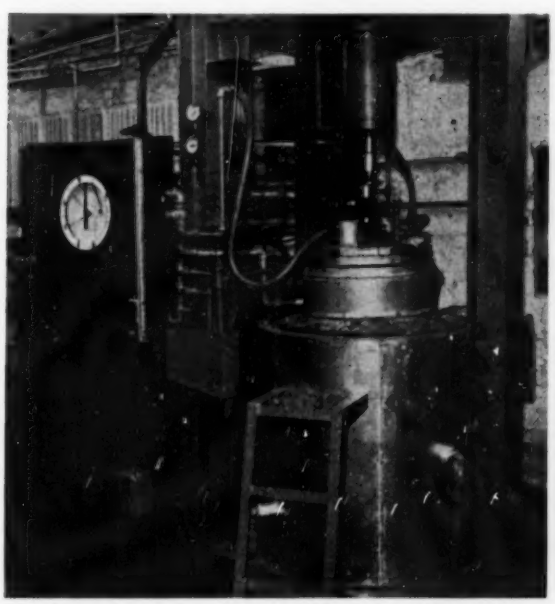
Charpy V notch specimens of Ti-Brush 40 were used in a series of tests with temperatures as a function.

ing may be performed after the initial sintering. Utilization factor is low, generally 1.7 lb of sponge being required to make 1 lb of titanium. The use of precision alloy steel dies assures close dimensional tolerances and minimizes machine labor, starting material costs and scrap loss. High density (4.52 g per cu cm) or controlled porosity may be obtained by this process. In order to attain a part of maximum density, a subsequent series of forming operations are necessary depending upon the design of the end product. Sintering or annealing operations are desirable between each pressing operation, the final operation being a stress relief anneal prior to finish machining.

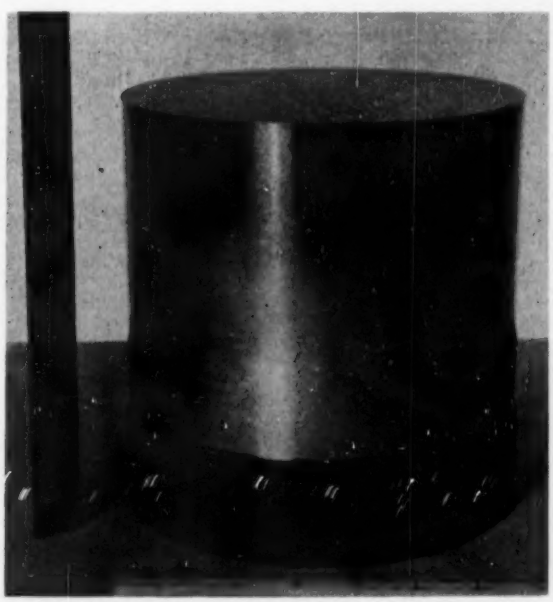
Mechanical Properties

The room temperature tensile properties of Ti-Brush 40, as the specific grade of Brush titanium with a 40,000 psi yield strength is called, were determined with a 0.25 in. diam tensile specimen having a 1 in. gage length. Typical room temperature mechanical properties are: 59,000 psi tensile strength, 40,000 psi 0.2 pct offset yield strength, 35 pct elongation, 53 pct reduction in area, 43 to 48 Rc A and a density of 4.50 per cu cm. These properties are comparable to those of melted and wrought titanium of similar compositions; however, as is well known, most powder metallurgy products are comparable in static strength properties but inferior in dynamic strength properties. For this reason, a detailed study of the fatigue and impact strengths of Ti-Brush 40 have been made.

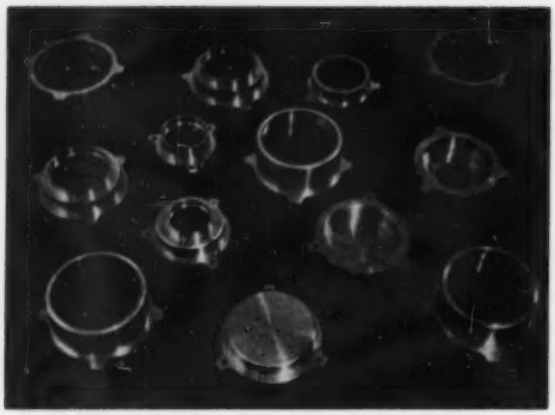
Notched and unnotched fatigue strengths of Ti-Brush 40 produced by both hot pressing and press forming were determined. These tests indicate that a fatigue strength of slightly over 30,000 psi at 10^7 cycles is realized by either method. Early reports on titanium powder metal products have indicated endurance ratios of 30 to 33 pct, but endurance ratios



The gas-fired vacuum sintering furnace is equipped with a hydraulic cylinder for hot pressing. High vacuum and temperatures prevent contamination and remove volatiles.



A 60 lb max density titanium ingot was produced by powder metallurgy. Lower section, which is unmachined, illustrates the limited amount of cleanup necessary.



Titanium bearing housings for jet engines are shown as machined. Housings were produced from prototype blanks made by hot pressing.

of 50 to 52.5 pct are consistently obtained using the fabricating techniques discussed here.

Since titanium is normally somewhat notch sensitive, having a notched endurance limit ranging from 15 to 25 pct, it was important to determine the effects of powder metallurgy fabrication on this vital design characteristic. Fatigue tests on notched specimens having a major diameter of 0.350 in., minor diameter 0.300 in., a 60° included angle notch and a notch root radius of 0.010 in. were performed at room temperature. The stress concentration factor on these specimens was 2.65, and the notched endurance limit was 23,000 psi at 10^7 cycles or 39 pct of ultimate tensile strength. The ratio of notched to unnotched endurance limits was about 1:3. Notch sensitivity of Ti-Brush 40 under the above specified conditions is approximately 0.18. All fatigue determinations were made on an R. R. Moore machine rotated at 10,000 rpm. Comparative ratios of fatigue strength to static strength for various metals are seen to be: Ti-Brush 40—0.39; Rc 70—0.38; 24S-T3—0.15; 75S-T6—0.15; and SAE 4340 HT 190—0.23. The specimens were not cooled in any way during the test.

Investigation of notch impact properties was conducted using Charpy V notch specimens. A series of tests were made as a function of temperature. It was seen that the impact energy remains relatively constant, between 36 and 41 ft lb, over a wide range of temperatures. Published data for unalloyed titanium have shown transition type curves indicating embrittlement at low temperatures apparently due to hydrogen. The processing methods used to produce Ti-Brush 40 maintain hydrogen at a low level preventing subsequent embrittlement. Since the rest of the impurities are also kept at a relatively low level, these effects on the notch impact strength are minimized.

Unalloyed titanium as processed has been evaluated by wet analytical methods and vacuum fusion techniques. The moderate tensile strength and high elongation achieved are probably attributable to the method of fabrication which does not permit the admission of gaseous impurities. A range of nominal analyses and maximum tolerable limits are shown in Table I.

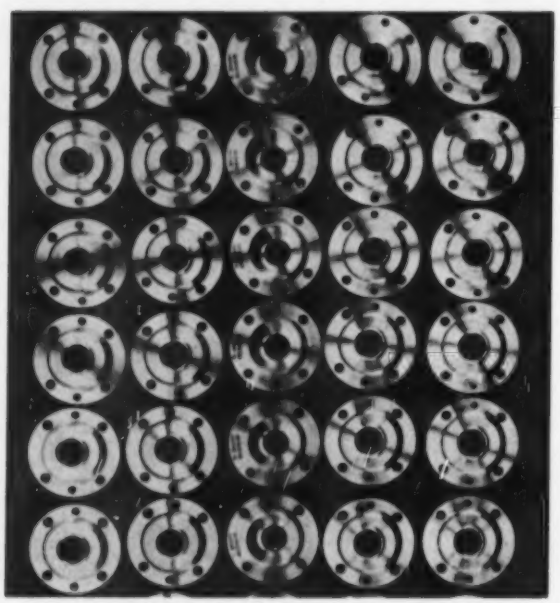
Summary

In view of the properties realized in both processes in notched and unnotched fatigue, energy absorption tests and tension tests, it seems logical to consider Ti-Brush 40 as fabricated by powder metallurgy as a significant material of construction. The low utilization factor realized through titanium powder metallurgy means savings—both in man power required for machining and materials required for production plus a low scrap loss.

In billet form, it appears comparable to arc melted stock for extrusion, drawing or forging. As a fabricated shape, its usefulness is enhanced by a high degree of uniformity characteristic of most powder metal parts.

Table I. Range of Analyses and Maximum Tolerance Limits For Ti-Brush 40, Pct

Element	Range	Maximum Tolerance (Tentative)
H	0.0003 to 0.0019	0.0050
N	0.013 to 0.016	0.020
O	0.090 to 0.110	0.150
C	0.010 to 0.050	0.050
Fe	0.080 to 0.150	0.250
Mg	0.010 to 0.05	0.10



Jet engine bearing housings produced by press forming show the reproducibility of the process.

Acknowledgment

The authors wish to thank the Engineering Services Dept., the Chemicophysical Dept., the Metallurgical Dept., particularly A. Hatch, R. Gridley, and A. D. Schwope, Director of Metallurgical Research, for the information given and cooperation rendered in preparing this report.

References

Relation Between Life Testing and Conventional Tests of Materials. *Bulletin* ASTM (March 1945).

Titanium. *ONR Report of Symposium* (Dec. 16, 1948).

Symposium on Titanium. *RDB 333/1* (Nov. 8 and 9, 1950).

Ultra High Strength Steel for Aircraft Structures. *Product Engineering* (October 1953).

Bifurcated Spouts Increase Open Hearth Output

Presented at the AIME National Open Hearth Steel Conference
Chicago, Apr. 5 to 7, 1954

Close Supervision Prevents Tieups

by Charles N. Straney

IN meeting the problems posed by tapping open hearth heats through bifurcated spouts into two ladles management at Weirton Steel Co. has found that close supervision is the best controlling factor. In actual operation, for exan.ple, tapholes must be kept straight and properly closed up. The hole must be dug and cleaned out before using the jet tapper.

Correct setting of runners, platform alignment, and stopper assembly of the dividing mechanism are necessary for good practice. Actually, when the point of the runner is centered and a good tap obtained, a large percentage of the heats will fill the ladle evenly without the use of the divider. The dividing mechanism is hand controlled and can be lowered into either runner at any point during the tapping period. Ten-in. clay brick is used on this rod. From Aug. 26, 1926, when No. 8 furnace tapped the first heat through a bifurcated spout, to Jan. 1, 1954, 23.8 million tons of steel have been tapped.

Bifurcated spouts have been found satisfactory for low carbon steels in Weirton's large furnaces. Results have been equally good on all grades and on structural steels of up to 0.30 carbon. The difficulties in making high carbon hot top steel, however, make it more practicable to use the single ladle, 280 ton furnaces.

C. N. STRANEY is Open Hearth Superintendent, Weirton Steel Co., Weirton, W. Va.

Pit space is always a problem and doubly so when open hearths have been enlarged to tap heats into two ladles, with crowding caused by each furnace having double ladle and thimble stands. Fifty-two ft of space are required for this setup at Weirton, thus cutting down the area between furnace ladle and stands normally used to prepare ladles for the next heat.

Ladles will always be a problem with bifurcated spouts because slag spouts must be on the right and left of the ladle. Extra space needed to set up the furnace properly cuts the area normally used for readying ladles for the next tap. Tapping heats of uniform size would aid the pit. Weirton has eight double-runner furnaces and five conventional types, and has been able to handle production with 26 ladles.

Good planning is required to prevent delays brought about by platform space needed to pour double ladle heats. Pouring platforms hold enough buggies to pour two ladles. Mold buggies are of standard gage design, with two stools and two mold lines. This kind of equipment cuts mold drags in half, saving platform space. Average length of the five platforms in the Weirton shop is 250 ft with 220 mold buggies in use most of the time.

At full operation, four 275 ton ladle cranes pour all heats. Two cranes handle miscellaneous work but are not used for heats. Close cooperation between open hearth and electrical departments keep crane delays at a minimum.

Shaped charge mounted on end of a cardboard tube is placed into open hearth furnace runway and against hard crust in furnace taphole at Weirton Steel Co. When detonated a jet of metal fragments is sent against the crust breaking it and starting the molten steel flowing.

Increased Shop Tonnages Realized

by E. Courtney Sorrells

AT U. S. Steel Corp.'s Gary Works tapping of heats through a bifurcated spout into two ladles has posed problems of equal division of heats, spout maintenance, and ladle availability. But with the difficulties has come an increase in the shop's maximum tonnage. The Gary Works has one furnace equipped to tap heats in this manner, with the first double heat tapped from No. 93 furnace in No. 5 open hearth Sept. 18, 1953. Average size of heat tapped has been 330 net tons.

No. 5 open hearth consists of eight stationary furnaces with a standard heat size of 185 tons. Over the past 18 months, six of the eight furnaces, including No. 93 furnace, have been converted to the extended hearth design. The hearth dimensions at sill line, inside brickwork, are 60 ft long x 16 ft wide, providing a hearth area of 960 sq ft. The bath is 36 in. deep at the center of the furnace. On the basis of a 330 ton heat, the hearth area per ton tapped is 2.9 sq ft.

Shop auxiliary equipment consists of three charging machines, two floor cranes, one 600-ton mixer, mixer crane, hot metal transfer car, three ladle cranes, eleven steel ladles, and three pouring platforms. Tapping of double heats has been accomplished without increasing auxiliary equipment.

All double heats are produced from a high scrap charge with approximately 35 pct hot metal. Oxygen is used for flame enrichment at the rate of 22,000 cu ft per hr, for 4 to 4½ hr during the meltdown and is also available for lancing. The product is low carbon rimmed steel for sheet and tin application. Charging time for cold stock is approximately 3½ hr with the hot metal addition within 5 to 5½ hr after begin charge. Tons per operating hour in the production of double heats averaged 35 pct higher than for other furnaces in the shop producing standard size heats. Fuel consumption averaged 12 pct lower. Ingot yield has been exceptionally high, averaging slightly over 90 pct.

Problems in controlling the tapping operation are principally confined to obtaining an equal division of the heat between the two ladles. Provided that the spout has been properly lined, with the branches of equal size, and the apex of the *nose* in alignment

E. C. SORRELLS is Division Superintendent, Steel Production, Gary Steel Works, U. S. Steel Corp., Gary, Ind.

with tap hole, an equal division of the heat can usually be obtained. A straight tap hole is also essential, and in this connection, the use of the jet tapper is of considerable help. However, if one ladle is filling more rapidly that the other and it becomes necessary to use a *divider* in an attempt to equalize the flow of metal into the ladles, the operation is usually not too successful. The *divider* or *diverter* as it may be called, consists of a 2 in. diam steel rod covered with a 10 in. diam clay stopper head and clay sleeves. The *divider* is suspended above the spout by means of a jib crane and manually positioned in the entrance of either branch of the runner with the aid of cables as it is lowered into position.

Considerable difficulty has been experienced in maintaining the bifurcated spout. The present practice is to line the spout with two flat courses of ladle brick laid-up with a high temperature air setting mortar. Replacement of the top course of brick, or working lining, is required after about 17 heats and replacement of the entire lining after approximately 50 heats. Considerable maintenance is necessary between relines. One castable lining was installed, but the lining life did not justify the additional cost.

Inasmuch as the product is low carbon rimmed steel variations in chemical analysis have not been a serious problem. Analysis variations have been confined to an occasional manganese spread between the first and second ladle and are practically always associated with an unequal division of the heat between the ladles. No particular difficulty has been experienced with manganese fading. Analysis variations have been responsible for the reapplication of the product of the second ladle on less than 5 pct of the heats.

Ladle availability is probably the most pressing pouring pit problem at the present time. The conversion of one furnace to double heats was made with no increase in the number of ladles. In addition, ladle outage time for relining has increased as the result of a decline in ladle lining life. Prior to tapping double heats, the average ladle lining life was 17 heats. The present lining life is 13 heats. This drop in lining life is apparently due to the large slag volume in the ladles resulting from tapping 165 tons of steel into 185 ton capacity ladles. Ladles are interchangeably used for both double and standard size heats. Experimentation is now in progress with nozzle pocket blocks, with outside insertion of the nozzle, which will permit faster cycling of ladles.

With only one furnace tapping double heats, there are no serious problems insofar as pit space is concerned. Slag and debris removal from the pitside area in front of the furnace does not present a problem since the center ladle base stand can be readily lifted-out for cleaning. The bifurcated spout must be cleaned and prepared in the pouring pit after each heat instead of on the tapping platform as is the case with the conventional straight spout.

Pouring platforms are not long enough to permit both ladles to be poured at one platform. Consequently, a double heat ties up two platforms for approximately one hour, leaving one platform and ladle crane to accommodate the remaining seven furnaces. Some delay is encountered when heats are bunched.

In conclusion, it can be stated that the conversion of one furnace to double heat in an eight furnace shop has made it possible to increase the shop's maximum tonnage by approximately 5 pct without the necessity of purchasing additional auxiliary equipment. A second furnace could probably be converted to double heats without any additional equipment with the possible exception of steel ladles. However, with only three ladles cranes, a sharp increase in furnace delays can be expected and the resulting tonnage increase from the conversion of a second furnace to double heats would probably be somewhat less than 5 pct.

Double Heats Show No Decrease in Quality

by J. H. Kelley

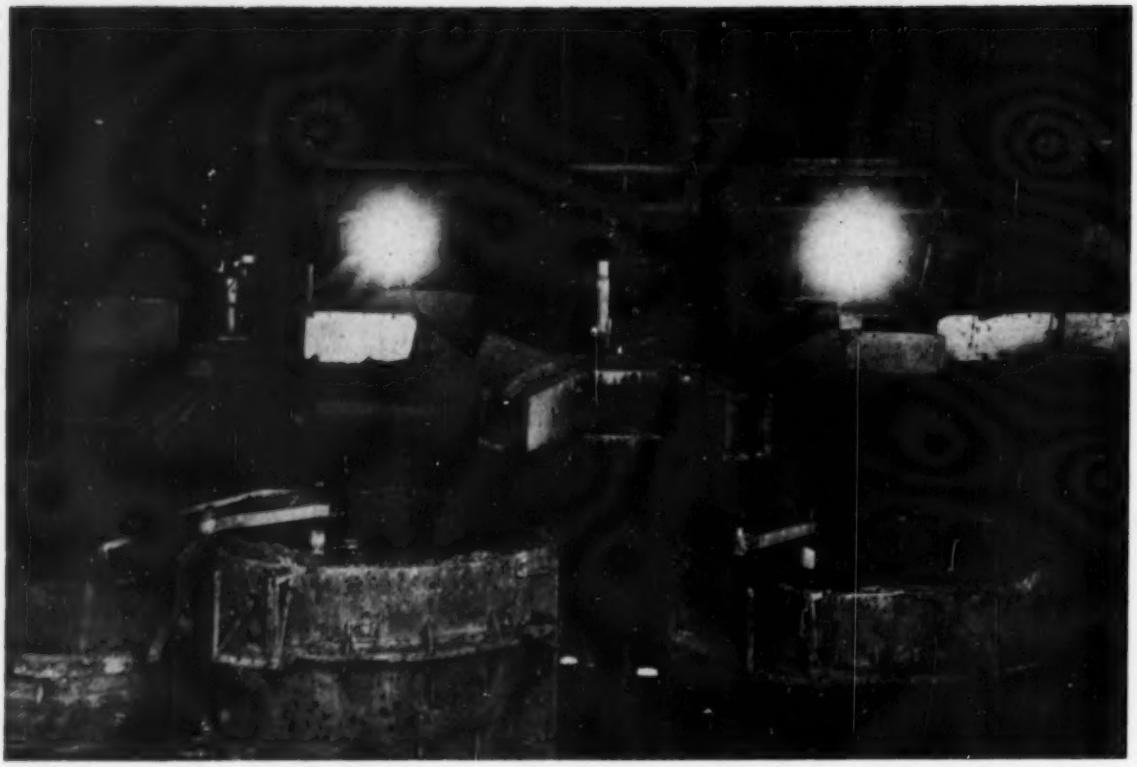
PROPER facilities, improved methods, and better fuel permitted the Sparrows Point plant of Bethlehem Steel Co., to go from single to double heats without decrease in quality.

No. 2 open hearth has given furnaces tapping 380 tons into two ladles. One furnace was converted from single to double heats in December 1951. The other four furnaces were converted in the latter part of 1953 and the first part of 1954.

J. H. KELLEY is Assistant Superintendent, Open Hearth and Bessemer Dept., Sparrows Point Plant, Bethlehem Steel Co., Sparrows Point, Md.

The burden on these furnaces can be either hot metal and scrap or young blown metal and scrap. The normal burden, at present, is hot metal and scrap as given in Table I. With this burden, each furnace is flushed through two slag notches in the back. No flush is taken when young blown metal is used.

The furnaces are fired with 65 to 70 pct high pressure coke oven gas and tar. The coke oven gas has from 50 to 100 grains of H_2S per 100 cu ft.



Open hearth furnace at Sparrows Point plant is shown with bifurcated spout and ladles in place. At present, double heats consist of low carbon rimmed and semikilled grades, but it is planned to make all carbon grades except those requiring hot topping.

During refining the furnace practice differs from single heats only in the rate of carbon removal. The rate of carbon drop on double heats is about 70 pct as fast as for a single heat. This, naturally, means a slower rate of oxidation and a little more care must therefore be taken with phosphorus control. Sulphur removal is not a problem because the fuel and hot metal are both low in this element. Furthermore, the hot metal is low in silicon.

Going to double heats has not increased problems in analysis variations. The double stock in back of the furnace is fed, rather than added in one batch, by means of an alloy chute. It is planned to make all carbon grades except those requiring hot topping. However, at present, only low carbon rimmed and semikilled grades are made in this shop.

Uniform analysis from ladle to ladle demands uniform filling of both ladles. With good tap hole and runner maintenance, the stopper rod for flow control is ample to insure both ladles filling to the same level. The center line of the stopper rod for flow control, must be set just a few inches beyond the dividing nose for the runner design used at Sparrows Point. There is seldom any difficulty with flow control. Electrical or mechanical failures in the stopper mechanism are the main cause for the infrequent difficulties encountered.

Table I. Normal Burden For 380-Ton Open Hearth Furnace

Material	Pct
Iron ore	8.0
Scrap	29.0
Cold metal	1.0
Hot metal	61.5
Alloys	0.5

Fluxes added are 120 lb and 10 lb per ton respectively of limestone and burnt lime.

No. 2 open hearth was originally built for duplex operation. Pouring pit space was provided throughout the length of both the open hearth and Bessemer building. Platform space will accommodate ingots from five ladles of open hearth steel. The Bessemer still is not poured in this area.

The pit space is sufficient to use and maintain 18 ladles and 10 bifurcated spouts. Thus, the pit space is ample for double heat operation. The only item eliminated was hot topping facilities. They were transferred to the other shops.

There are three 290-ton ladle cranes and one 40-ton auxiliary crane. The third 200-ton ladle crane was installed to insure the ladle cranes available at all times. The 40-ton crane is used for ladle and runner maintenance.

Additional pouring problems may be encountered by the double moves required to take the ladles away once the heat is tapped. Delays, or false moves, will increase the time—tap to open—which will affect pouring practice. Giving this phase the required attention, it has been possible to maintain the normal percent dry shut off (98 pct or better).

Cold heats and pouring troubles are definitely related. The Pt-PtRh thermocouple is used to control tapping temperatures. Furthermore, the excellent heat transfer from firing tar and high pressure coke oven gas has helped to go from single to double heats without any increase in pounds of skull per ton of steel.

Brick Sleeve Solves Tapping Problem

by H. H. Northrup

IN the Buffalo plant open hearth of Republic Steel Corp. the design and strength of ladle crane runways make it impractical to carry more than 130 tons of steel per ladle.

In 1937 a program was started to enlarge two of the nine furnaces, equip them with double ladle stands, and bifurcated spouts with flow regulating devices. This doubled the furnace capacity but presented steel quality, pouring, and delivery problems. These difficulties were met and overcome and three more furnaces enlarged, and equipped with bifurcated spouts in 1939, 1945, and 1951. The shop presently consists of five furnaces with bifurcated spouts averaging 250 tons per heat, and four furnaces with single spouts averaging 125 tons per heat.

The first of the many problems to be worked out with the installation of bifurcated spouts was one of accurately dividing the stream to fill both ladles evenly to prevent excessive pit scrap and off chemistry. Experience showed that a 10-in. diam brick sleeve placed slightly ahead of the division point of the spout would divert the stream into one leg or the other, provided the following items are checked prior to tapping:

A—That the width of each leg of the runner inside of brick lining at the point of division is held at 14 in. when lined. This allows a 12-in. opening when 1-in. of slurry has been added to each side of the spout between heats.

B—That the position of the divider is checked before tapping to be sure it will fall into the proper position to deflect the flow of steel as desired when lowered during tap. The action of the divider or flow regulator is one of deflecting the stream just before it gets to the point of division rather than plugging one or the other of the spout legs.

Positioning of the flow regulator in the runner while tapping is achieved by the use of an adjustable arm mounted above the taphole on a shaft connected to a remotely controlled wheel and gear. The flow regulator arm is hinged where it meets the control shaft so that this arm may be pushed from one side of the runner to the other to divert the stream of metal to the ladle desired. If the previously indicated checking is properly carried out with adjustments if necessary, it is possible to divert almost the entire stream to either leg of the spout, During the tap, the melter watches the rise of metal in the ladles from an elevated platform at one end of the furnace and gives instructions for placing the flow regulator to the crew below him. Experience in distribution of metal has been excellent and it is seldom that there is more than 10 tons difference in weight of the two ladles. Any difference can usually be attributed to difference in ladle capacities arising from wear on ladle lining.

H. H. NORTHRUP is Superintendent, Open Hearth Dept., Buffalo Plant, Republic Steel Corp., Buffalo.

For a short time after the installation of bifurcated spouts, production on these furnaces was confined to reinforcing bar steel. It was thought that higher quality steels could not be successfully produced. Gradually this was proved wrong and for the past five years the size of the order has been the only determining factor in scheduling the furnace. In periods or reduced operation all grades of steel will be made in furnaces with bifurcated spouts.

Space for ladle additions is limited to one side of the ladle because of the bifurcated spout. Most of the production at present is killed steel in the medium manganese range, thus most of the additions, except silicon and aluminum, are normally made in the furnace. Should grades of steel produced change to those requiring larger amounts of ladle additions, chutes to handle this material can easily be installed.

Manganese additions for semi-killed or rimmed steels with the usually low manganese specification are shoveled into the ladle. This results in little variation between the two ladles, not too surprising when it is considered that a 10 pct difference in ingot weight on a 30 point addition would mean a variation of only three points from the mean.

Ladle additions of an exothermic nature must be limited because of fumes produced, which, depending on the speed of tap, obscure the height of steel in the ladles from the melter.

With five of nine furnaces of bifurcated design, the shop is commonly referred to as having a nine-furnace charging floor and a 14 furnace pit. To adequately handle the product of all furnaces and keep two ladles off for relining there are 17 all welded, 130-ton ladles. With recent results of a new practice of setting nozzles from the outside, it is believed that it will be possible to maintain the same production with a fewer number of ladles in service.

Four ladle cranes are sufficient usually, to handle the furnaces ready to tap. It is important that the pit foreman communicate with the senior melter many times during the shift. By planning in this manner, melters have been able to change grades of steel scheduled from one furnace to another to avoid heats tapping so close together that there is delay-waiting on ladle cranes. Planned weekly repair turns on ladle cranes are a *must* and breakdowns are thus at a minimum.

Because the three pouring platforms will hold only sufficient molds for the contents of seven ladles, it is apparent that at full operation each heat could not be held at the platform for the usual 80 min after finish pour. This problem was tackled with the help of the metallurgical dept. and it was found that if each half of the heat was pulled out of the pit within 5 min after finish pour and then allowed to stand outside until time to strip the hot top, the internal quality of the ingot was not adversely affected on most grades of killed steel.

Transportation Factors Are Key to Development of Caribbean Bauxite For U. S. Aluminum Industry

by Arthur F. Johnson

THE principal costs of making a pound of aluminum are for 9 kw-hr of electricity and for 1.9 lb of the oxide (Al_2O_3) called alumina. A pound of alumina is made by digesting 2 or 3 lb of bauxite in hot caustic solutions, settling and filtering out the oxides of silicon, iron, and titanium and precipitating and calcining the Al_2O_3 as a pure, white powder. The cost of foreign bauxite delivered at an alumina plant in the U.S. may equal or exceed the cost of its chemical treatment to make alumina.

Some of the principal factors affecting costs in the utilization of foreign bauxites are: transportation from mine to market, mineralogical and physical composition of the bauxite, thickness of the bauxite beds and their clay and forest overburden, amenability of the bauxite to beneficiation and to caustic extraction of the alumina, and the size of bauxite deposits and rate of mining employed.

Most foreign bauxite used in North America is mined in British or Dutch Guiana or Jamaica. In Jamaica in 1952, 404,755 long tons of bauxite were mined by three producers (Aluminium Ltd., Reynolds, and Kaiser) and 264,988 long tons imported into U.S. apparently all for Reynolds plants. About 1.3 to 1.6 million tons were imported into the U.S. by Kaiser and Reynolds in 1953. Exports of alumina from Jamaica to Norway in the first quarter of 1953 were about 5000 tons with declared value of about $363,000.

Exports of bauxite to the U.S. from Surinam in 1952 were 3,109,375 long tons and apparently were about the same in 1953. In 1951 about 80 pct was Alcoa bauxite production and 20 pct Billiton production. At the end of 1951 Alcoa's Moengo Hill mine was exhausted after producing 10.25 million tons in 25 years and a new mine 9 miles east was being opened. In British Guiana the Demerara Bauxite Co. (Alcan) exported 1,968,190 tons of dried bauxite in 1952. Table I gives the analyses of bauxite from various regions.

Transportation Cost and Aluminum Cost

Transportation from mine to market is a major cost component. It becomes most important considering that the ultimate market center for fabricated aluminum metal in the U.S. probably lies in Indiana or Ohio, and that the bauxite from the time it is stripped and mined is merely being processed in transit. Bauxite may be beneficiated in Surinam, chemically treated to make alumina in Mobile, electrolytically reduced to make the metal aluminum with cheaply sold hydropower in Washington, and rolled into sheet at Davenport, Iowa, before final shipment to destination in the U.S. market centers further east. The transportation route from mine to market may cover 7000 miles or more. Rather than analyze any one particular set of costs, if a certain bauxite and the metal made from it travels a particular route such as that given above, it was chosen to break transportation costs down to costs per ton mile by truck, ship, and railroad since this enables one to make comparative estimates with various routings.

Fig. 1 is a graphic comparison of the costs per ton mile vs length of haul. Table II shows approximate cost range of mining and beneficiating bauxite, making alumina, and reducing it to aluminum pig. Total costs for each type of operation are equal to the operating costs shown plus a percentage of the investment cost to provide for amortization and interest. This might amount to 8 pct when normal amortization is 20 years. An aluminum producer's overall costs consist of those shown in Table II plus the costs of transportation from one type of plant to another. To save freight on alumina, reduction plants have been built on the Gulf Coast although heavy investments in power plants were required.

Bauxite Transportation

Handling bauxite by truck in the Caribbean countries is now restricted to hauling a mile or a few miles from the mines to the treatment plant. In British Guiana at the principal bauxite mines along the Demerara River a 36-in. gage railroad with 60-lb rail has been used from the mines to a wash-

Table I. Analyses of Bauxite From Various Regions, Pct

Location	Al_2O_3	SiO_2	TiO_2	Fe_2O_3	H_2O
Jamaica	50.65	1.09	2.52	19.53	26.2
African Gold Coast	49.00	1.00	—	22.00	—
Surinam	57.8	2.90*	2.40*	5.20	—
British Guiana	57.8*	2.90*	2.40*	5.20*	—

* Estimated.

A. F. JOHNSON is associated with the Metals Div., Olin Industries Inc., East Alton, Ill. This article is an abstract of the paper presented at the AIME Annual Meeting, New York, Feb. 15 to 18, 1954.

ing plant. Since some mines are now about 40 miles distant, heavier rail is now being installed. At the mines on the Berbice River, bauxite is trucked a mile or less to a washing plant after which the washed bauxite is barged about 138 miles down river to the drying plant. Ocean vessels with draft less than 18 ft can pick it up at the ship loading point shown in Fig. 2.

In Jamaica, Aluminium Ltd. makes alumina at the mine to eliminate all bauxite hauling, except a truck or scraper haul of a mile or even less. The alumina is hauled via a Government railroad about 35 miles to the coast where it can be loaded on ships. Reynolds uses an aerial tram shown in Fig. 3 to haul the dried bauxite 6 miles from the mines to the coast. Kaiser uses a 13-mile standard gage railroad, 70 ton gondolas, and 1200-hp diesel-electric locomotive to haul bauxite to a drying plant on the coast.

Trucking Costs

Trucking is flexible transportation and competitive with narrow and standard gage railroads for short hauls particularly where care is taken to build good roadbeds which determine speeds as well as repair and maintenance costs. In the final transportation of aluminum from the fabricating plant to market, trucks are carrying an important part of the product because they speed delivery. Some fabricators have their own trucks and deliver to points 1000 miles or more away. An interesting new development is the loading of truck trailers, one or two per flat car, on the New York, New Haven & Hartford RR. This combination affords the flexibility of the truck with the lower cost of the railroad.

Since component items in trucking costs vary from place to place and from one year to another, data is given only as an example of cost computation. It brings out that repair and maintenance and fuel are radically reduced on a good roadbed where higher speed attained also reduces labor cost and illustrates that turn around time is important.

Regularly scheduled hauling by truck on U.S. paved highways can be done for 3¢ per ton mile and occasionally as low as 2¢ per ton mile, with a payload in both directions. Bauxite hauling costs in foreign countries must be corrected for the fact that truck parts will cost 10 to 25 pct or more, depending on freight and import duties. Skilled labor in the Caribbean may be as low as $14 per 40-hr week but lower productivity, more supervision, and subsidies to the mining camp community may reduce the advantage in labor cost to about one third or one half U.S. labor costs per ton mile.

Railroading Costs

Actual cost of most hauling by rail in the U.S. probably ranges from 3¢ per ton mile to 0.5¢ or less including capital charges as well as operating costs. Costs are less with regularly scheduled payload in both directions, or on long prairie hauls, and rise with less-than-carload lots, with payload in only one direction, or on short mountain hauls.

Using diesel locomotives, costs per train mile will be about 2.4 times the cost per locomotive mile. Average total costs per train mile can be converted to gross ton mile costs by dividing by the average 2900 gross tons hauled, and to costs per net ton mile by dividing by 1300 net tons hauled. From this, diesel haulage in the U.S. costs about 0.1¢ per ton mile, excluding enginemen's wages and handling of empties. For switching, the diesel is even more economical than steam. Capital cost for a 180-ton diesel locomotive can be amortized for about 0.05¢ per ton mile. Repair and maintenance of cars costs less than 0.1¢ per ton mile.

In the U.S. a railroad track will cost $30,000 to $75,000 per mile, depending whether the rail is 90-lb used rail or 132-lb new rail with heavier ballast. The expense of roadbed preparation may double this cost. A sample of how such capital cost is distributed is shown for standard gage roadbed near Pittsburgh costing $53,568 per mile of track. Track must be amortized in 30 years even when repair and

Table II. Operating and Investment Costs in Making Aluminum Pig from Bauxite

Operation	Cost in Cents Per Lb of Al Produced*	Investment in Cents Per Lb of Al Per Year
Bauxite mining and beneficiation	0.5 to 1.1	2 to 6
Alumina manufacture	1.5 to 2.5	11 to 15
Reduction of alumina	7 to 10	50 to 60**

* Operating costs exclude amortization or interest on the investment.
** Includes capital cost of gas or coal fired power plant.

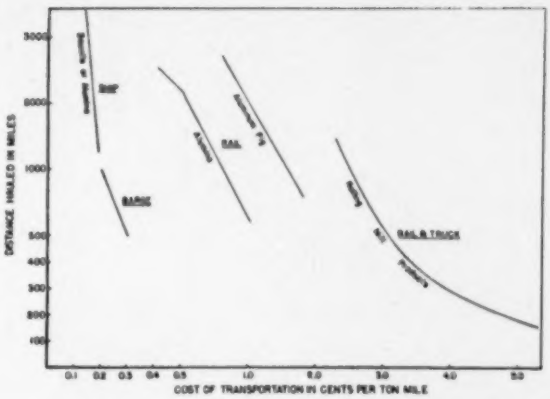


Fig. 1—A comparison of the costs per ton mile vs length of haul is shown. The transportation route from mine to market for bauxite and aluminum may cover 7000 miles or more.

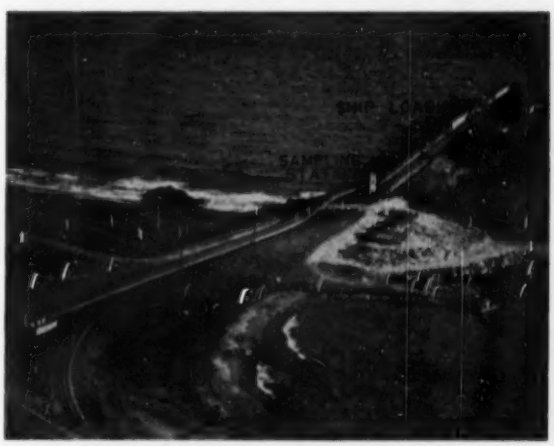


Fig. 2—Sampling station and ship loading point serve Kaiser's Jamaica bauxite operation. Port and railroad to mines inland represented part of capital cost of opening up deposit.

Fig. 3—Towers being erected here now serve aerial tramway transporting bauxite from mine to Ocho Rios Bay, Jamaica.

maintenance expense in 30 years equals the amortization expense. It requires a comparatively heavy traffic of two trainloads of 60 cars every day of the year to reduce roadbed amortization plus repair and maintenance cost below 0.23¢ per ton mile so it must be considered a major cost of railroad transportation.

Ocean Shipping Costs

Shipping costs vary radically according to the supply of ships. The past year costs have been dropping because there are more ships available and less demand for space. At present some charters are available for little, if any, more than ship operating costs; and bauxite can be hauled from Guiana or Surinam via Trinidad to the U.S. for about $6.00 to $7.00 per long ton plus U.S. port charges of about $1.00.

Ton mile costs are increased by the turn around time. Hauling from Jamaica to Kitimat, about 10,000 miles round trip, it would require perhaps 23 days steaming time at sea plus 3 in port and 2 going through the Panama Canal making a total of 28. The unit costs per long ton mile must accordingly be increased from the above per day in service by roughly 20 pct. Alcan's shipping arm Sagterms of Montreal has chartered a standard Victory ship S.S. Sun Karen and altered her to carry 12,000 long tons alumina at a service speed of 14 knots. Another new vessel S.S. Sunrip will carry 12,600 long tons at 13.5 knots.

It is about 2500 miles round trip from Jamaica to New Orleans, 6000 miles round trip from the Guianas to New Orleans, and 6900 miles round trip from the Guianas to Arvida, P.Q. Shipment from the Guianas is complicated by the fact that ships with a draft of more than about 19 ft cannot get over the rivermouth bars into the Guiana ports. For instance, 4000 to 6000-ton vessels are used to shuttle from the Guianas to Trinidad where they are unloaded into land storage. Bauxite is reloaded into a 10,000-ton or larger vessel plying between Trinidad and the U.S. gulf ports or Arvida. The 10,000-ton vessel can take 45 to 50 pct of its load in the Guianas and complete the cargo by stopping off at Trinidad. On the return to the Guiana port the ship may carry 4000 tons of bunker C oil for drying bauxite there. In spite of the transfer charges involved, the haul can be contracted for about $6 to $7 per ton from Guiana to Gulf ports. The trend is towards larger quicker unloading vessels to save time and stevedore charges in port. Reynolds' ship named after the late Carl Schmedeman, AIME member, is a 13,000-ton capacity ship plying between Jamaica and Corpus Christi and is reported to be making the 2000-mile round trip in about 9 days with loading and unloading time of only 8 hr each. The quick unloading is done by a self-contained belt unloading system on the ship. Alcoa is reported building a 32,000 and a 26,000-ton vessel as well as an 8,000-ton ship for shuttle service. The crew of the 26,000-ton ship is estimated as 51, speed 12.75 knots with 5800 hp, and cost $5 million built in Norway.

Such ships can be built cheaper abroad although U.S. machinery is generally preferred. Cost of a hull plus machinery in Japan is perhaps $150 per deadweight ton compared to $300 in the U.S. Wage costs are about three times as much with U.S. crew under the U.S. flag as with British, Dutch, or Norwegian crews under their respective flags.

Barging Costs

Barging has had an interesting history in its application to freight movement of bauxite. It forms a cheap means of freight movement in South America and Africa where railroads inland have not been built and still offers competition to U.S. railroads in the Mississippi River Valley and coastal areas. For Mississippi River barge traffic a 9-ft deep channel is necessary to compete with other forms of transportation, and 14 to 16 barges with 1300-ton capacity each may be moved by one tug. Barging can be contracted for a cost of about 0.25¢ per ton mile of river distance. In the shallow and narrow upper portions of the South American rivers two or three 400-ton capacity barges have been towed by one tug.

From the foregoing analyses of transportation costs for foreign bauxite it is easy to compute that the freight on a ton of foreign bauxite to its alumina plant is much greater than the value of the bauxite at the mine, unless the alumina plant happens to be located at the mine. This is the case of Aluminum Ltd.'s plant in Jamaica and becomes economic because no import duty on alumina to Canada is involved and process fuel would cost as much in Canada as in Jamaica. The characteristics of the ore both physically and chemically also make location of the alumina plant in Jamaica advantageous.

Conclusion

From the long range viewpoint foreign bauxite cost will rise gradually with increasing wage rates and taxes excepting where producers are able to counteract such increases by improved handling methods. Domestic bauxitic clays and anorthosites may ultimately replace foreign bauxites as a source of aluminum. Although treatment costs are greater on domestic ores, transportation costs between plants are greatly reduced.

Exclusive of the cost of bauxite, operating costs of making alumina may run from $15 to $25 per ton of alumina produced. This operation cost is made up chiefly of labor, repair, and maintenance, 12 to 14 million Btu of fuel and the caustic soda lost in the red mud. The cost of the 2 or 3 tons of bauxite needed may exceed the total treatment costs when stripping, mining, beneficiation, and transportation to the alumina plant are all considered. Capital charges for the alumina plant, the mining plant and ships to carry the bauxite add to the total cost of production.

On a production-per-day basis, the Steubenville Works of Wheeling Steel Corp. produces an average of 4333 tons of steel which requires 12.1 billion Btu's from fuel tar, coke oven gas, and fuel oil sources.

Fuel Balance at Steubenville Works Controlled by Utilizing Summarizers

by John H. Dickson and Grover C. Nuzum

OPEN hearth operations at Wheeling Steel Corp.'s Steubenville works demand 12,133,324,000 Btu per day. Fuels available to the open hearths are coke oven gas, fuel tar, and fuel oil. Two of the fuels, the gas and fuel oil, are coke oven byproducts.

The gas yield from the coke ovens is approximately 11,300 cu ft per ton of coal charged. The virgin tar yield is about 8.5 gal per ton of coal charged or, translated to the 20 pct cut of virgin tar, a ton of coal yields about 6.8 gal of fuel tar.

J. H. DICKSON and G. C. NUZUM are Field Engineers, Utilities Dept., Steubenville North Works, Wheeling Steel Corp., Steubenville, Ohio.

On the basis of 6900 tons of coal charged per day a yield of approximately 46,920 gal of fuel tar per day is available for open hearth consumption, and approximately 77.97 million cu ft of coke gas is available for underfiring the ovens and distribution to other consumers.

Of the total gas production 40 pct or 31.188 million cu ft is used for underfiring the ovens. Approximately 1.5 pct or 1.170 million cu ft of the total production is bled. The balance or 45.612 million cu ft is distributed as shown in Table I.

The gas consigned to the open hearth and annealing departments is washed with sodium carbonate

Open hearth liquid fuel pumping equipment is shown. Combinations of liquid fuels have been used in proportions of 1:1 and 3:2 fuel oil and fuel tar. Fuels can be controlled individually or in combination by utilizing a summarizer based on total heat output.

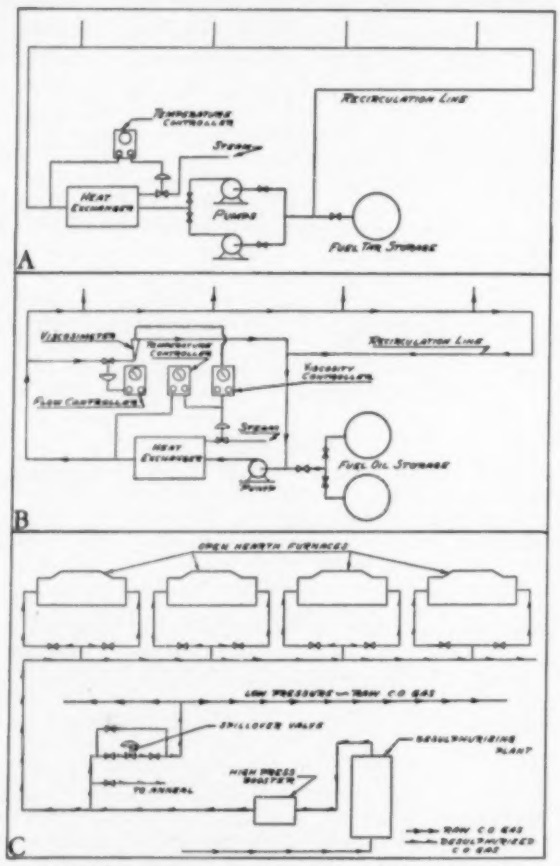
solution in a hot vacuum process desulphurizing plant to reduce sulphur content from 450 grains per 100 cu ft to 100 grains per 100 cu ft. To minimize sulphur pickup in steel maximum allowable sulphur in gas for the open hearth is 100 grains per 100 cu ft.

Open Hearth Fuel

Table II gives the analyses for coke oven gas, fuel tar, and fuel oil. The 10 million cu ft of coke oven gas assigned to open hearth use is desulphurized and delivered to the open hearth at a 13 psi gage. To insure sufficient line pressure a flow slightly in excess of normal consumption is pumped. Excess gas is released by a flow regulated spillover valve, dumping the high pressure desulphurized gas into low pressure raw coke gas system.

Table I. Coke Oven Gas Distribution

Area	Cu Ft of Gas (1000)
Coke Plant	
Boilers	500
Sintering plant	500
Steubenville North	
Open hearth furnaces	10,000
Open hearth mixer and misc	2,000
Reheating furnaces	7,700
Soaking pits	4,500
Anneal	3,800
Boilers	1,000
Blast furnace and misc	1,200
Galvanize	500
Steubenville South	
Soaking pits	1,800
Boilers	1,000
Bottom ovens	1,000
Metal mixer	500
Blast furnace and misc	1,200
Yorkville and Beechbottom	5,800
Line loss and unaccounted for	2,612
Total	45,612



Circulation systems for various kinds of fuel used at the Steubenville Works open hearth are: A—fuel tar system, B—fuel oil system, and C—coke oven gas system.

Table II. Analyses of Fuels Used in Open Hearth Furnace, Pct

Type	C	H	O	N	S	Free C*	Residue	Ash	CO_2
Fuel tar**	92.54	4.85	0.80	1.09	0.55	17.59	—	0.15	—
Coke oven gas***	—	53.50	0.20	4.40	—	—	—	—	2.3
Fuel oil	88.99	9.99	0.22	0.21	0.55	99.90	0.04	—	—

* High molecular weight hydrocarbons insoluble in CS_2.
** Contains 0.02 pct H_2O.
*** Contains 6.9, 29.3, and 3.4 pct respectively of CO, methane, and illuminants.

Fuel tar is transported to the open hearth at temperatures between 350° and 400°F in insulated tank cars and stored in a 124,000 gal capacity insulated tank. The storage tank is equipped with steam heating coils to maintain temperature; however, it has never been necessary to heat the storage tank.

To supply the open hearth furnaces the fuel tar is circulated through the shop at 275 psi gage by two steam driven reciprocating piston pumps. A thermal electric heating system, used on the fuel tar lines to prevent temperature losses, maintains a line temperature of 300°F from the circulating loop to the furnaces. Maximum burner pressure is 215 psi gage at 300°F.

Fuel oil is pumped into two 124,000 gal capacity storage tanks for open hearth consumption. Steam heating is also available on the fuel oil storage tanks but is seldom used. The fuel oil is circulated through the open hearth at a pressure of 300 psi gage at 190°F. Maximum burner pressure is 230 psig at 170°F. A heat exchanger is controlled by a continuous viscosimeter or temperature control to maintain the proper oil viscosity. A return line to the suction side of the pumps completes the circulation loop.

Fuel tar and oil are atomized by steam controlled from a panel board by a manual air loading valve. Steam is maintained at a line temperature of 590°F, and 250 psi gage. Maximum burner pressure is 120 psi gage at 450°F. Combustion oxygen line pressure is maintained at 47 psi gage.

Open Hearth Control

Various fuels can be controlled individually or in combination by utilizing a summarizer based on total heat input. Because of detrimental flame characteristics at low flows of coke gas a minimum firing rate has been set at 35,000 cu ft per hr. Combinations of liquid fuels have been used in proportions of 1:1 and 3:2 fuel oil and fuel tar. Any amount of coke gas burned in the furnace automatically cuts the liquid fuel or fuels according to the Btu input of the gas.

Combustion air is controlled through a summarizer, and use of combustion oxygen produces a decrease in combustion air. Maximum combustion oxygen consumption is 25,000 cu ft per hr. In normal practice 10 pct excess air is used, with seasonal changes in ambient air temperatures bringing variations.

Table III. Daily Requirement of Open Hearth Fuel Utilizing Maximum Gas Consumption

Fuel Per Day	Btu (1000)	Pct
Coke oven gas—7,660,000 cu ft*	3,745,740	30.87
Fuel tar—46,920 gal**	7,694,880	63.42
Fuel oil—4,810 gal***	692,704	5.71
Total	12,133,324	100.00

* 489 Btu per cu ft. ** 164,000 Btu per gal. *** 144,000 Btu per gal.

Pump house and storage tanks service the open hearth. White tank is the 124,000 gal insulated fuel tar tank and is equipped with steam heating.

Open Hearth Heat Balance

Normal operations at the Steubenville Works open hearth result in a production of 130,000 tons of steel per month. Using a 62 pct hot metal charge, the average fuel rate is 2.8 million Btu per ton. Converted to a production-per-day basis, 4333 tons of steel require 12,133,324,000 Btu from fuel tar, coke oven gas, and fuel oil sources.

The entire production of fuel tar is consigned to the open hearth. Therefore, 46,920 gal of fuel tar with a calorific value of 164,000 Btu per gal will supply 7,694,880,000 Btu of the total daily heat requirements. The balance of 4,438,444,000 Btu per day can be supplied with 9.077 million cu ft of the 10 million cu ft of coke oven gas available for open hearth consumption.

To date the best month for coke gas consumption in the open hearth averaged 7.66 million cu ft per day. On this basis the open hearth fuel requirements of 12,133,324,000 Btu per day were supplied with fuel tar, coke oven gas, and fuel oil in the proportions given in Table III.

Coke oven gas at the Steubenville works has a net heating value of 489 Btu per cu ft. At present it is not possible to fire the full available daily coke oven gas volume because of lack of sufficient draft. The general practice is to fire 80,000 cu ft of gas per hr per furnace before hot metal. After hot metal the firing rate is reduced to 35,000 cu ft per hr.

It Took The Mesabi Discovery To Beat Edison's Taconite Venture

THOMAS Alva Edison, the Wizard of Menlo Park, wagered $1.5 million on an idea that didn't pay off but that might have if he had lived long enough. Edison believed that low grade magnetic iron ores could be utilized, and during the late 1880's and 1890's put up three quarters of what eventually amounted to a $2 million investment in his magnetic separator and Ogden mine in northern New Jersey.

While Edison wasn't the only inventor to try magnetic separation, his activities at Llewellyn Park, N. J., and at Ogden were singular. Several innovations that were later to become standard mining practice were born at Ogden.

When Edison decided to launch the magnetic ore project, the existence of suitable deposits was not too well known. Because they were considered economically worthless, magnetic iron ore deposits were not recorded. Edison had designed a dipping needle. With needle in hand his men crossed and recrossed the country between the St. Lawrence and the Potomac. When a body of magnetic ore was discovered the ground was retraversed at intervals of 100 ft.

Edison and his associates acquired by purchase or lease some 16,000 acres. The Ogden property was one of the largest of the ore bearing areas. The extent of the orebody was developed by trenching every 100 ft across the strike of the deposit and taking samples at regular, short intervals. Tests indicated that ore averaged about 20 pct iron.

Edison departed radically from traditional crushing methods. Instead of screening after every crushing operation, material passed through a series of machines without any attempt at sizing until actual separation began. Edison quarried, mined, and recrushed the ore to what was then considered fine size, then separated magnetic iron from nonmagnetic gangue, mixed the concentrate with a binder, pressed the product into briquettes, dried it in a furnace, and loaded into cars for less than 6¢ per unit of the iron content of the briquettes. E. Gybbon Spilsbury, commenting on the process, said:

". . . until the price of foundry pig shall advance to a figure at which it will be profitable to purchase 68 pct iron-ore at 6¢ a unit, it is not probable that the Edison works can be run continuously at a profit. It stands, nevertheless, a monument of perseverance in original research which certainly deserves our admiration."

The rock passed through a series of crushers until they came to the 14-mesh screen. The "giant" rolls were 6-ft diam, 6-ft face, and placed 7 ft 3½ in. apart on centers. The rock went from the "giant" rolls to intermediate rolls. After a third set of rolls the rock went to a dryer. Crushing machines were driven by a horizontal compound engine, rated at 700 hp, using steam at 150 psi. Two Climax type boilers supplied the steam. After screening, the magnetic separator invented by Edison took over. The separator was simple and cheap to operate.

THOMAS ALVA EDISON

"When a thin stream of material is allowed to fall by a stationary magnet, those particles which are subject to magnetic attraction are deflected from their path toward the magnet. Two parallel streams of material are formed, one behind the other, which may be readily collected in different receptacles."

One of the most important facets of Edison's Ogden mine operation was the necessity for keeping costs at a minimum. Conveyor belts had been used to transport grain, but before Edison employed them at Ogden, they had never been adapted for ore handling. The first belts used at Ogden were 20 to 30 in. wide, and as long as 500 ft between centers. Ore abrasion wore them out quickly. Repair and renewal bills were high. That was the situation when Thomas Robins, Jr., visited the mine for the first time. He noticed that the thin layer of rubber which covered the belt resisted the abrasion much longer than did a corresponding thickness of cotton duck which formed the body of the belt. Each layer or ply of duck wore more quickly than the preceding one. Wear was greatest in a line along the center of the belt.

Robins had a belt made with a thicker layer of rubber on the carrying side. It was also thicker in the center. Edison was quick to go along with the idea and experiments at the mine were successful. Eventually, using the mine as a kind of laboratory, Robins developed an improved mechanism for carrying the belt. It consisted of a set of rollers or idlers arranged on angle brackets, raising the sides of the belt to form a trough. It eliminated the need for guide-boards and increased belt life.

Edison pushed experiments in concentration and separation at Ogden as long as it appeared that work would lead to an economic process. One day the inevitable had to be faced. Mesabi range discoveries of tremendous reserves of high grade iron ore meant the end of low grade concentration. ***M. A. Matzkin***

Methods for Separating Rare-Earth Elements in Quantity

As Developed at Iowa State College

by F. H. Spedding and J. E. Powell

WHILE rare earths are reported to be widely distributed in nature and are not really rare,* in practice, there are only a few minerals which are sufficiently rich in rare earths to serve as practical sources. Perhaps the best known of these is monazite which is a phosphate mineral containing rare earths and thorium. This mineral occurs as a dense brown sand in gravel beds and is particularly rich in the light rare earths of the cerium subgroup. This mineral is processed commercially for its thorium, cerium, and lanthanum content, and, consequently, furnishes rich concentrates from which neodymium, praseodymium, samarium, europium, and gadolinium may be obtained. Unfortunately, monazite is rather lean in rare earths heavier than gadolinium. A second mineral which is rich in the light rare earths is bastnasite, a fluorocarbonate. Extensive deposits of this ore have been discovered in the western United States and have received considerable newspaper publicity in recent years. While bastnasite is very rich with respect to cerium, lanthanum, and neodymium, it contains even less heavy rare earths than does monazite.

* Geologists estimate that cerium and yttrium are more abundant than many of the more familiar elements such as zinc, tin, mercury, silver, etc.

One of the better sources of heavy rare earths of the yttrium subgroup is gadolinite, a black silicate rock from which the rare-earth content can be extracted readily by acid leaching. It is obtained chiefly from Norway at the present time, although there are known deposits in the United States. Other sources of heavy rare earths include fergusonite, euxenite, and samarskite which are refractory tantalo-columbate ores. These minerals require caustic fusion or reduction to carbides with carbon before the rare-earth content can be extracted. All of the minerals which are rich in the heavy rare earths contain yttrium as a major constituent.

After the rare earths have been extracted as a group from an ore by chemical means, it is generally convenient to precipitate them from acid media with oxalic acid in order to eliminate certain non-rare-earth impurities such as iron, beryllium, etc., which are usually present. The oxalate can then be readily ignited to R_2O_3. The oxide can be dissolved in acid and is the starting point for subsequent separation into the pure components.

F. H. SPEDDING and J. E. POWELL are associated with the Institute for Atomic Research and Dept. of Chemistry, Iowa State College, Ames, Iowa.

Discussion on this paper, TP 3880D, may be sent, 2 copies, to AIME by Dec. 1, 1954. Manuscript, Feb. 11, 1954. New York Meeting, February 1954.

Perhaps the principal reason why the rare earths have not been studied as extensively as other elements of the periodic table, whose natural abundances are comparable, is that they are extremely difficult to separate from each other by the usual chemical means. Prior to 1945, the separation of one trivalent rare earth from another was a laborious process. All separations were based on repeated fractionation processes, i.e., fractional precipitation, fractional decomposition, fractional crystallization, etc. These processes were repeated from a few hundred to many thousands of times in order to obtain individual rare-earth salts of reasonable purity. Of course, mention should be made that, in the few cases where a rare earth could be oxidized or reduced to a valence state other than three, more conventional chemical means could be utilized to separate the oxidized or reduced ion from the other normally trivalent rare earths. The ionic states which deserve special mention are Ce^{IV}, Sm^{II}, Eu^{II}, and Yb^{II}. When it is possible to remove an element of the series efficiently, due to an optional valence state, its immediate neighbors also become easier to isolate. For example, binary mixtures of lanthanum and cerium, and praseodymium and cerium can be obtained by a relatively small number of fractional operations. The tetravalent state of cerium then allows the complete resolution of the binary mixtures by ordinary chemical means. Although the tetravalent state of cerium has been known for a long time, the divalent states of samarium, europium, and ytterbium were not used extensively in separations prior to 1930 because they are relatively unstable in aqueous media.[1-11]

No attempt will be made to give a comprehensive review of the extensive literature dealing with the separation of rare earths. Rather, this paper will be confined to a review of those methods which have been developed at Iowa State College during recent years, and which have proved extraordinarily successful for the isolation of highly pure rare earths in quantity.

It was obvious that, if pure rare earths were to become generally available, methods would have to be developed wherein the thousands of fractional operations made necessary by the similarity of rare-earth properties could be performed automatically. The development of chromatographic techniques and ion-exchange resins appeared to offer a mechanism by which this objective could be accomplished.

A number of early attempts were made to separate rare earths by these means; for example, Russell and Pearce[12] passed a mixture of rare earths through a cation-exchange column and reported

The Transactions papers appearing in the Journal of Metals during 1954 will subsequently appear in AIME Transactions Volume 200, and may be permanently referenced to that volume.

some enrichment of individual rare earths in the effluent solution. Erätmetsä, Sahama, and Kanula[18] used columns of activated alumina with similar results and also reported that, when the rare earths were complexed with citrate, the order of enrichment was reversed. While these investigators obtained some enrichment, their results were not sufficiently promising to lead to further intensive investigation or to the production of pure rare earths in quantity.

Since early 1945, three distinct processes involving ion-exchange resins have been developed at Ames which have been exceptionally efficient in producing quantities of individual rare earths in a high state of purity. They are:

1—The elution of an adsorbed band of mixed rare earths down a cation-exchange bed in the hydrogen or ammonium state by means of a 5 pct citric-acid solution, adjusted to a pH of 2.5 to 3.0 with ammonium hydroxide.

2—The elution of an adsorbed band down a cation-exchange resin bed in the hydrogen state with a 0.1 pct citric-acid solution, adjusted to a pH of 5.0 to 8.0 with ammonium hydroxide.

3—The elution of an adsorbed band of rare earths through a cation-exchange resin bed in the copper II cycle with an ammonium salt of ethylenediaminetetraacetic acid.

In all of the above methods, which have been used in the Ames laboratories for the successful separation of rare earths, a commercial-grade sulphonated styrene-divinylbenzene copolymer was used. Resins of this type are sold under the trade names Nalcite HCR, Dowex-50, Amberlite IR-120, and Permutit Q. They consist essentially of sulphonated benzene nuclei which are linked and cross-linked by means of aliphatic chains.

The hydrogen ion of the sulphonic-acid group can be readily replaced by other cations. If hydrogen is attached to these groups, the resin is said to be in the hydrogen state and, similarly, if ammonium ion or rare-earth ion is attached, the resin is in the ammonium or rare-earth state.

The polymeric network of the organic portion of the exchanger results in a porous structure, throughout which solvent and neutral molecules can migrate freely. Positive ions can also diffuse through the resin readily as long as electrical neutrality is maintained. Whenever a positively charged ion enters the resin lattice, it is necessary that an anion accompany it or that another positive ion leave the resin phase. If the solution outside the resin is of low ionic strength, negative ions are effectively prevented from diffusing into the resin by the high concentration of negative sulphonic groups attached to the lattice. Each gram of dry resin has a fixed number of these anionic groups attached to it, depending upon the method of manufacture. If the ionic strength of the external solution is low, the resin will adsorb an equivalent number of positive charges whether they are borne by hydrogen, ammonium, rare earth, or any other cation.

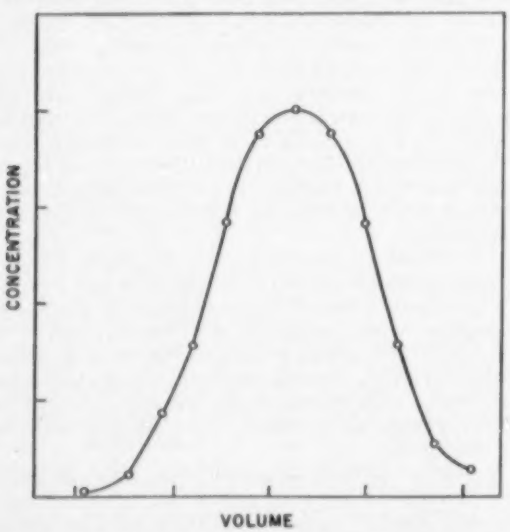


Fig. 1—Bell-shaped elution curve obtained with concentrated citrate at a low pH.

Since it is desirable to maintain uniform band fronts during elution, the commercial-grade resin is sieved in order to obtain resin particles which are reasonably uniform in size. A quantity of the air-dried resin is saturated with water and poured into glass columns which have either a porous plate or a metallic screen to support the resin bed. The size and shape of these columns are determined by the amount of rare earth to be processed, but, in the laboratory, the resin beds are generally about 4 ft long and from 1 to 15 cm in diameter. If longer beds are desired, a number of the short columns can be used in series.

The resin beds are backwashed with water prior to use in order to eliminate any fine particles which were not removed by screening and to obtain a uniformly packed resin bed. After the beds have resettled, the resin is converted to the desired cationic state by passing an excess of a solution of the cation through the resin. The excess solution is rinsed on through the column with water.

The mixed rare-earth oxides are dissolved in hydrochloric acid and a known quantity of this solution is poured into the column, the amount depends upon the length of adsorbed band desired and the diameter of the bed. Since the influent solution contains cations of only one type (mixed rare earths), these cations completely replace the cations they encounter as they enter the top of the bed, and a saturated band of rare earths with a relatively sharp front is formed at the top of the column. Below the band front, the resin remains in its original state. Since the affinity of the resin for rare-earth ions differs very little with the species involved, very little separation occurs in the loading step.

Next, a solution of some negative-ion species which forms a tight complex with the rare earths is passed downflow through the column. This sets up a competition for the rare-earth ions between the aqueous and resin phases. Since the columns are operated so that equilibrium for this competition is maintained, an individual rare-earth ion continually exchanges between the complexing ion and the resin. The positive ions in the complexing solution replace the rare-earth ions at the rear edge of the band so that the rare-earth band is driven down the resin bed. Because the stability constants of the rare-earth complexes differ slightly from one rare earth to another, the most tightly complexed rare earth moves most rapidly down the column. The behavior of the band during its passage down the column, the efficiency of separation of the individual rare-earth species, the quantities which may be handled, etc., differ in the three methods referred to above.

The first method was developed at Ames during World War II[14, 15] and simultaneously and independently at Oak Ridge.[16-20] In this method a 5 pct (or similar concentration) of citric acid, adjusted to a pH of 2.5 to 3.0 with ammonia, is used as the eluant.**

** Somewhat higher citrate concentrations can be used if the solutions are adjusted to slightly lower pH's and, conversely, with somewhat lower concentrations, slightly higher pH values can be used.

Here, the adsorbed rare-earth band spreads out as it progresses down the column and continues to spread as long as the band is moving through the resin bed. Analyses of the resin show that the mol fraction of rare earth to ammonia constantly varies throughout the adsorbed band. Behind the adsorbed band the rare earth is completely replaced with eluant cations, since the resin comes in contact only with these cations, and whatever rare earths are liberated are immediately swept downstream by the flowing solution. Once the band has moved far enough down a column so that it is not affected by the original saturation effect, it is found that the band contains very little rare earth at the back; the concentration of rare earth reaches a maximum near the middle and diminishes to a low value again at the front. As the adsorbed band of a rare earth moves off the bottom of a column, a bell-shaped elution curve (volume vs concentration) is observed, see Fig. 1. The longer the column through which a band is eluted, the flatter the bell-shaped curve will be, since the ordinate will be lower and the abscissa broader. The ammonium ion of the eluant constantly overrides the rare-earth front; in practice, elution could have been started equally well with an ammonium-state resin bed instead of a hydrogen resin bed. The individual rare-earth bands travel at different rates under a given set of conditions so that their maxima get progressively farther apart as the elution progresses. The overlapping of one band into another may be considerable; so, unless the adsorbed band travels many times its original length, high purity individual rare earths cannot be obtained in good yields. While the above behavior has made this method highly desirable for separating minute or tracer quantities of rare earths, it is not competitive with methods 2 and 3 for isolating large quantities of pure rare earths, nor does it usually result in as high purities.

In the second method, the rare-earth band is eluted down a column with 0.1 pct citric-acid solution, adjusted to a pH between 5.0 and 8.0 with ammonium hydroxide.[24, 25] In practice, values near 8.0 are preferable, since the concentrations of rare earths in the eluate are greater. Consequently, the higher pH results in larger yields per unit time. Under these conditions, the rare-earth band spreads out initially as it moves down a resin bed until it reaches an equilibrium value. From there on, the sharp front edge of the band progesses down the column at the same rate as the sharp rear edge, see Fig. 2. The elution curve is rectangular in shape rather than bell-shaped. Under these conditions, it is found that the concentrations of the total rare earth, the ammonium ion, and the hydrogen ion in contact with any part of this band are constant (see Fig. 3) and that the ratio of concentrations between these constituents is the same in the eluate as on the resin in contact with it. In this method, the ammonium ion of the eluant does not overrun the front edge of the adsorbed rare-earth band; therefore, only pure hydrogen-state resin is found ahead of the

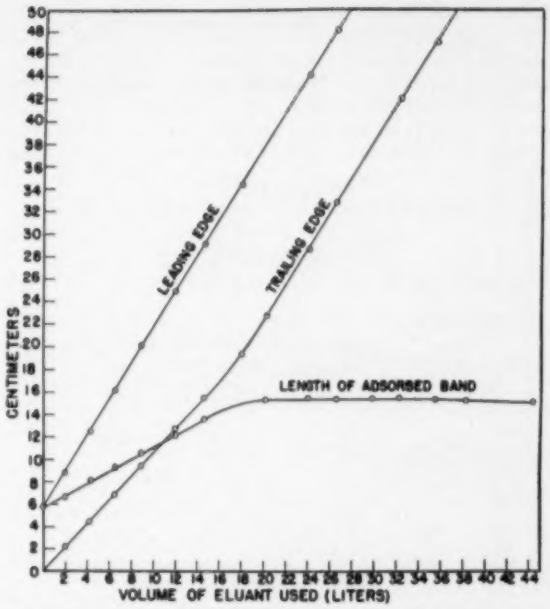


Fig. 2—The development of a typical adsorbed rare-earth band on a Nalcite HCR bed under the influence of 0.1 pct citrate eluant.

adsorbed band. Throughout the band of an individual rare earth, there exists a uniform composition of ion species. The hydrogen-ion concentration is very low compared to the concentrations of ammonium ion and total rare earth, both on the resin and in the eluate. Following the equilibrium band, the resin is, for all practical purposes, entirely in the ammonium state. Around a pH of 8.0, the equilibrium rare-earth band is approximately double the length of the original adsorbed band. At lower pH values, the equilibrium length becomes longer, approaching infinite length in the neighborhood of a pH of 5—the exact pH depends upon the rare-earth species. If several rare-earth species are present, they tend to separate into individual bands which ride head to tail down the column with the heaviest rare earth leading. The boundaries between the individual species are extremely sharp and it should be emphasized that the bands do not pull apart from each other no matter how far they are eluted.

The theory which accounts for the formation of these bands is extremely simple in principle although the solution of the algebraic expressions requires some labor. There are about 12 important unknown variables in the system, but it is possible to write 12 independent relationships involving only material balances, electrical neutrality, stability constants of the various complex ions formed, and equilibrium conditions. Therefore, if the composition of the eluant, the capacity of the resin bed, and the total amount of rare earths adsorbed on the bed is known, it is possible to calculate the concentrations of all ions in the eluate and on the resin to better than 1 pct accuracy and to predict the rate of movement and the equilibrium lengths of the adsorbed bands on the column. While in theory nearly 100 pct yields of pure individual rare earths should be obtained one after another, in practice, there is always some overlap in the elution curves where binary rare-earth mixtures are obtained. This results from the fact that it is virtually impossible to avoid small amounts of channeling, some

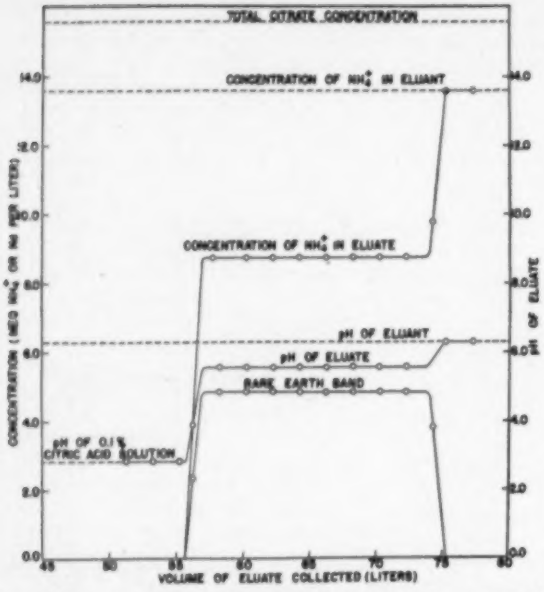

TOTAL CITRATE CONCENTRATION
CONCENTRATION OF NH4+ IN ELUANT
CONCENTRATION OF NH4+ IN ELUATE
pH OF ELUANT
pH OF ELUATE
RARE EARTH BAND
pH OF 0.1 % CITRIC ACID SOLUTION
CONCENTRATION (MEQ NH4+ OR M PER LITER)
pH OF ELUATE
VOLUME OF ELUATE COLLECTED (LITERS)

Fig. 3—A typical elution curve obtained by eluting an adsorbed rare-earth band from a Nalcite HCR bed with 0.1 pct citrate under equilibrium conditions.

tilting of the bands, and some nonequilibrium conditions which are related to the size of the resin particles and the flow rate of the solution past them. The overlap can be minimized by choosing the diameter of the columns so the lengths of the equilibrium bands of individual species will be long compared to the diameter of the resin bed and by using uniform fine resin particles in the beds and appropriate flow rates.

It follows from theory that, since the concentration of $Cit^{=}$ ion in equilibrium with an individual band is predetermined by the composition of the eluant and will be somewhat different for each species due to differences in the stabilities of the complex ions, the equilibrium conditions will tend to give self-sharpening boundaries. For example, if a praseodymium ion gets into the neodymium band, the $Cit^{=}$ concentration found there will be too low and it will necessarily move more slowly down the bed than the neodymium ions until it is overtaken by the main praseodymium band. Conversely, a neodymium ion in the praseodymium band will be subjected to so high a $Cit^{=}$ concentration that it will move more rapidly than the praseodymium ions until it overtakes the main neodymium band. This behavior can also be used to advantage in resolving binary mixtures which result from channeling and tilting of bands. The binary rare-earth solution flowing from a column where channeling and tilting has occurred, instead of being treated with oxalic acid to recover the rare-earth content, can be simply adsorbed on the top of another column. If similar binary mixtures from parallel-column runs are also adsorbed on this column to give a band of sufficient length, the rare earths will re-form into individual bands upon elution, with only a single region of overlap.

Operations of this type have been scaled up to a unit which utilizes 45 6-in. and 100 4-in. columns with beds 4 ft long. It is possible to load these columns with about 50 lb of mixed rare earths every 10 to 15 days. The eluant flows through columns by gravity with very little attention, and most of the effluent citric acid is recycled. Most of the rare earths are recoverd in such a state that, by simple chemical procedures, yields of 40 to 90 pct of individual rare earths, 99 to 99.99 pct pure, are obtained. The binary mixtures which result have to be reworked. It should be mentioned that, since the main objective of the laboratory personnel is research and not production, the columns are not operated at this rate continually, but only during periods of intensified effort.

Under the conditions usually employed, there are certain adjacent rare earths which do not form sharp bands on columns of reasonable length. They do, however, separate partially. The binary band for one of these pairs usually has a composition which varies from 95 pct of one rare earth to 95 pct of the other at opposite ends of the band. This results from the fact that the difference between the stability constants of the citrate complexes of these adjacent rare earths is not as large as the average difference between the stability constants for adjacent rare earths.

One of the difficult pairs to separate is ytterbium and lutetium. Fortunately, ytterbium forms divalent salts while lutetium does not, and, since ytterbium and lutetium readily separate from thulium and the other rare earths by ion exchange, these rare earths can be obtained in a pure state by extracting the ytterbium from aqueous solution into sodium amalgam according to the method of Marsh,[6] or by electrolytic reduction after the method of Yntema.[3, 5]

The fact that the stability constant for the ytterbium-citrate complex lies closer to lutetium than to thulium is probably related to the fact that ytterbium will form divalent salts. Similarly, separation is poor between gadolinium, europium, and samarium. It is well known that samarium and europium both form divalent salts. The methods used to separate ytterbium from lutetium can be used to separate both samarium and europium from gadolinium. The europium can then be separated conveniently from samarium by selective reduction of the europium with zinc according to the method of McCoy.[5, 4]

While yttrium is not a true rare earth, the stability constant for its citrate complex falls between the constants for dysprosium and terbium. Since yttrium is always a major constituent of any natural heavy rare-earth concentrate, it is difficult to separate terbium and dysprosium from this element. However, by eluting the mixture through longer columns, or a series of short columns, a separation can be obtained between dysprosium and yttrium. Although terbium can be greatly enriched, yttrium trails badly into the terbium fractions. Perhaps the best method for purifying terbium is method 3.

Method 3 is, in principle, similar to method 2. Here, however, the rare-earth band is eluted with an ammonium salt of ethylenediaminetetraacetic acid (EDTA). While this method has not been investigated to the extent that the citrate methods have, it is already evident from preliminary experiments that this method is more desirable and economical than the citrate methods for separating the more difficult groups of rare earths. It shows exceptional promise even for the light rare earths, La through Nd, and the more readily separable heavy rare earths. The stability constants of the rare earth-EDTA complexes are much further apart

than the rare earth-citrate complexes[28] and, therefore, the tendency to form equilibrium bands is more pronounced. In preliminary experiments, the resin bed of one column is completely saturated with a mixture of rare earths from a chloride solution. The chloride ion and excess rare earth are washed out of the column with water and the adsorbed band is eluted down a second column containing resin in the copper II state with an eluant comprised of an ammonium salt of EDTA in water. As the EDTA solution moves over the rare-earth bed, the rare-earth ions are picked up as their EDTA complexes and ammonium ion is deposited leaving an ammonium resin bed behind, as in the citrate process. When the rare earth-EDTA eluate comes in contact with the copper bed, the rare earths are deposited, and the stronger copper-EDTA complex then flows out of the column over the copper-saturated resin bed. The copper ion plays a role analogous to the hydrogen ion in the citrate process, since in competing for the EDTA it causes the rare earths to deposit, forming a sharp boundary. Of course, other ions which form tight soluble complexes with EDTA can be used in place of copper. It is required that the ion species used eluate ahead of the rare earths being separated. This will happen if the stability constants of their complexes with EDTA are greater than those of the rare earths being eluted. However, as in the case of copper II, if the valence of the positive ion itself is less than three, the EDTA stability constant may be somewhat less than the heavier members of the rare-earth series. The relative affinities of the adsorbed ions for the resin are also involved and trivalent ions are more firmly held by the resin than divalent or monovalent ions, in general.

The optimum conditions for EDTA elution of rare earths have not yet been determined, although the method works quite well for an eluant containing 5 to 20 pct EDTA in the pH range around 7 or 8.

The method has the distinct advantages that even the most difficult groups separate much better with EDTA than with citrate and the volume of eluant required is only a fraction of that needed for an efficient citrate elution.

It is interesting to note that, if rare earths, which are not adjacent and occur several numbers apart in the series, are to be separated, this can be done quite rapidly by taking advantage of the relatively large differences in the stability constants of their complexes with EDTA. Thus, if just enough EDTA is added to a 50-50 mixture of Nd^{+3} and Er^{+3} to the theoretically complex Er^{+3} present, and then the mixture is poured through a thin bed of resin sufficient to adsorb at least the quantity of rare earths not complexed by EDTA, a marked enrichment takes place.[27] The resulting filtrate will contain 95 pct of the total Er, while the resin retains 95 pct of the neodymium originally present. Two or three applications of this process will produce highly pure specimens of both Nd and Er.

The Ames Laboratory has done considerable work on solvent extraction as a means of separating rare earths,[28] as have others.[29, 30] However, in no case has anyone developed a solvent-extraction method starting with a natural rare-earth mixture, wherein adjacent rare earths have been separated in sufficient quantities to serve as a comparison with ion-exchange methods. While the solvent-extraction method holds considerable promise for economically separating the light rare earths in tonnage quantities, a great deal of work needs to be done before this can be demonstrated. It is the authors' opinion that the existing methods of liquid-liquid extraction are not likely to compete economically with ion-exchange methods for the separation of extremely pure individual rare earths if only a few hundred pounds are to be produced.

Acknowledgment

Work was performed in the Ames Laboratory of the Atomic Energy Commission.

References

[1] L. F. Yntema: *Journal* American Chemical Society (1930) **52**, p. 2782.

[2] R. W. Ball and L. F. Yntema: *Journal* American Chemical Society (1930) **52**, p. 4264.

[3] H. N. McCoy: *Journal* American Chemical Society (1935) **57**, p. 1756.

[4] H. N. McCoy: *Journal* American Chemical Society (1936) **58**, p. 1577.

[5] P. W. Selwood: *Journal* American Chemical Society (1935) **57**, p. 1145.

[6] D. W. Pearce, C. R. Naeser, and B. S. Hopkins: Electrochemical Society (1936) **69**, p. 557.

[7] J. K. Marsh: *Journal* Chemical Society (1937) p. 1367.

[8] J. K. Marsh: *Journal* Chemical Society (1942) pp. 398, 523.

[9] J. K. Marsh: *Journal* Chemical Society (1943) p. 8.

[10] J. K. Marsh: *Journal* Chemical Society (1943) p. 531.

[11] T. Moeller and H. E. Kremers: *Industrial Engineering Chemistry* (1945) **17**, Analytical Ed., p. 798.

[12] R. G. Russell and D. W. Pearce: *Journal* American Chemical Society (1943) **65**, p. 595.

[13] O. Erätmetsä, T. G. Sahama, and V. Kanula: *Ann. Acad. Sci. Fennicae* (1951) **A57**, No. 3, pp. 5-20.

[14] F. H. Spedding, A. F. Voight, E. M. Gladrow, and N. R. Sleight: *Journal* American Chemical Society (1947) **69**, p. 2777.

[15] F. H. Spedding, A. F. Voight, E. M. Gladrow, N. R. Sleight, J. E. Powell, J. M. Wright, T. A. Butler, and P. Figard: *Journal* American Chemical Society (1947) **69**, p. 2786.

[16] E. R. Tompkins, J. X. Kyhm, and W. E. Cohn: *Journal* American Chemical Society (1947) **69**, p. 2769.

[17] J. A. Marinsky, L. E. Glendenin, and C. D. Coryell: *Journal* American Chemical Society (1947) **69**, p. 2781.

[18] D. H. Harris and E. R. Tompkins: *Journal* American Chemical Society (1947) **69**, p. 2792.

[19] B. H. Ketelle and G. E. Boyd: *Journal* American Chemical Society (1947) **69**, p. 2800.

[20] G. E. Boyd, J. Schubert, and A. W. Adamson: *Journal* American Chemical Society (1947) **69**, p. 2818.

[21] E. R. Tompkins and S. W. Mayer: *Journal* American Chemical Society (1947) **69**, p. 2859.

[22] S. W. Mayer and E. R. Tompkins: *Journal* American Chemical Society (1947) **69**, p. 2866.

[23] E. R. Tompkins, D. H. Harris, and J. X. Kyhm: *Journal* American Chemical Society (1949) **69**, p. 2504.

[24] F. H. Spedding, E. I. Fulmer, J. E. Powell, and T. A. Butler: *Journal* American Chemical Society (1950) **72**, p. 2354.

[25] F. H. Spedding, E. I. Fulmer, J. E. Powell, T. A. Butler, and I. S. Yaffe: *Journal* American Chemical Society (1951) **73**, p. 4840.

[26] E. J. Wheelwright, F. H. Spedding, and G. Schwarzenbach: *Journal* American Chemical Society (1953) **75**, p. 4196.

[27] E. J. Wheelwright: Unpublished data.

[28] J. H. Bochinski: Unpublished Doctorate thesis submitted to Iowa State College. (1954) Iowa State College Library, Ames, Iowa.

[29] D. F. Peppard, J. R. Farris, P. R. Gray, and G. W. Mason: Argonne National Laboratory, Lemont, Ill. (1952) AECD-3327, ANL-WMM-927.

[30] B. Weaver, F. A. Kappelman, and A. C. Topp: Oak Ridge National Laboratory, Oak Ridge, Tenn. (1952) ORNL-1408.

Slag-Metal-Graphite Reactions and the Activity Of Silica in Lime-Alumina-Silica Slags

by James C. Fulton and John Chipman

Reduction of silicon from blast-furnace-type slags by carbon-saturated iron is a very slow reaction even under conditions of rapid stirring. Equilibrium under atmospheric pressure of carbon monoxide was approached from both sides, and the silica-silicon relation was established at temperatures of 1425° to 1700°C for slags containing up to 20 pct Al_2O_3.

Silicon carbide is formed by reaction of graphite with high silica slags and the conditions for its formation were determined.

Activities of SiO_2 and of CaO in their binary solutions at 1600°C were determined. The effects on the activity of SiO_2 were established for additions of 10 to 20 pct Al_2O_3 and of 10 pct MgO.

STUDIES of chemical reactions between blast-furnace slags and metal have been focussed on the transfer of sulphur from metal to slag. The important role which silica reduction plays in this process[1] has led to a study of the transfer of silicon from slag to metal and the equilibrium represented by the equation

$$SiO_2 \text{ (in slag)} + 2C \text{ (graphite)} = \underline{Si} + 2CO \text{ (g)}. \quad [1]$$

The authors[2] have published an account of the experimental method employed in attacking this problem and of three series of experimental results at 1600°C. This paper contains data on some additional compositions and extends the observations to cover the temperature range 1425° to 1700°C. The data are used to determine the activity of silica in the slags.

As in the earlier experiments, the atmosphere was carbon monoxide which flowed through the furnace at 150 ml per min. In certain heats designated in the tables and figures, the gas was bubbled through the melt using a graphite tube. The latter increased in porosity during a run and in some cases bubbling ceased as indicated by cessation of surging. Some results were discarded for this reason.

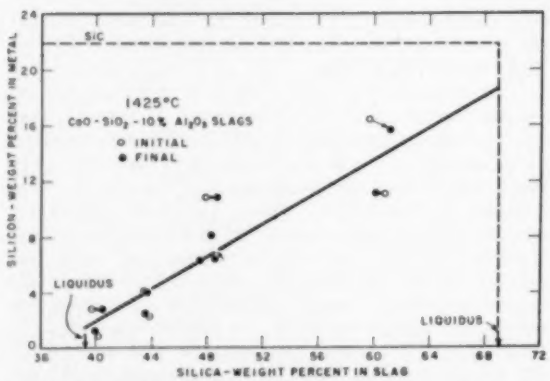


Fig. 1—Silicon distribution for Fe-Si-C alloys in equilibrium with CaO-SiO_2-10 pct Al_2O_3 slags under 1 atm of carbon monoxide.

Experimental Results

The new data are shown in Tables I through V and both old and new data are plotted in Figs. 1

J. C. FULTON, Junior Member AIME, is associated with Allegheny Ludlum Steel Corp., Brackenridge, Pa., and J. CHIPMAN, Member AIME, is Professor of Metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

Discussion on this paper, TP 3821C, may be sent, 2 copies, to AIME by Dec. 1, 1954. Manuscript, Apr. 4, 1954. New York Meeting, February 1954.

This paper is based on a thesis by J. C. Fulton submitted, in partial fulfillment of the requirements for the degree of Doctor of Science, to Massachusetts Institute of Technology.

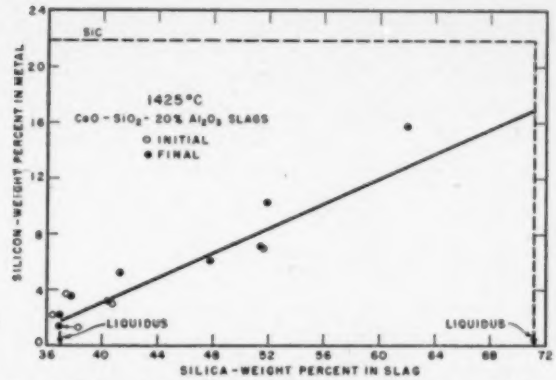


Fig. 2—Silicon distribution at 1425°C for Fe-Si-C alloys saturated with graphite and in equilibrium with $CaO-SiO_2$-20 pct Al_2O_3 slags under 1 atm of carbon monoxide.

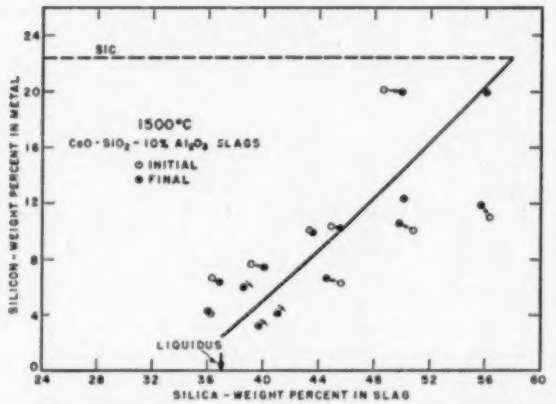


Fig. 3—Silicon distribution at 1500°C for Fe-Si-C alloys saturated with graphite and in equilibrium with $CaO-SiO_2$-10 pct Al_2O_3 slags under 1 atm of carbon monoxide.

through 10. Minor changes have been made in the locations of some of the curves previously published.

Carbon in the Slag: In plotting the data, a correction for the dilution effect of carbon entrapped in the slag was applied to the silica analyses when carbon was determined. The two main sources of carbon in the slag were: 1—graphite particles released from the iron bath or from the small globules of graphite-saturated iron entrapped in the slag sample and 2—particles from the easily abraded carbo-cell that was used as an insulating material above the crucible. In view of the large number of analyses, it was decided to determine the carbon content only when the analysis was definitive with respect to the equilibrium curves.

It was found that visibly clear glassy slags contained less than 0.10 pct C. Darker samples contained as much as 2.0 pct. In certain cases, the necessity for carbon analysis was avoided by igniting the pulverized slag in oxygen before weighing the sample, but this was successful only in the more refractory slags.

Silicon Monoxide in the Slag: Silicon carbide was found dispersed as small crystals in a few slags taken from melts which were initially higher than 23 pct Si. In such cases, the apparent analysis, when silicon is reported as SiO_2, totals more than 100 pct. This anomaly in slags from ferrosilicon containing 45 to 75 pct Si was ascribed by Gel'd and Esin[9] to the

Table I. Samples from Heats Made at 1425°C

Heat & Sample No.	Time at Temperature, Hr	Metal, Wt Pct Si	Slag Analyses, Wt Pct SiO_2	CaO	Al_2O_3	C	Wt Slag/ Wt Metal (Grams)
126	Charge	4.0	44.0	45.5	10.5	—	400/400
−1	1.0	4.13	43.56	—	—	0.09	
−3	5.0	4.12	—	—	—	—	
−5	8.0	4.07	43.73	—	10.72	—	
130	Charge	1.0	40.0	50.4	9.6	—	400/400
−1	1.0	0.91	40.15	—	—	0.08	
−2	2.0	1.12	—	—	—	—	
−5	8.0	1.31	39.94	—	10.94	—	
133	Charge	6.0	50.0	40.0	10.0	—	500/400
−1	1.0	6.36	48.61	—	—	0.07	
−2	2.0	6.34	—	—	—	—	
−5	8.0	6.48	48.59	—	11.21	—	
143	Charge	2.5	43.7	45.6	10.7	**	450/570
−1	0.5	2.39	43.83	—	—	0.09	
−2	1.0	2.41	—	—	—	—	
−6	8.0	2.53	43.53	—	10.60	—	
144	Charge	3.0	40.0	49.1	10.9	**	360/450
−1	0.5	2.90	39.65	—	—	0.21	
−2	1.0	2.91	40.20	—	—	—	
−6	8.0	2.87	40.47	—	8.73*	—	
145	Charge	8.0	48.6	40.9	10.5	**	405/475
−1	0.5	8.17*	48.00	—	—	0.23	
−2	1.0	8.22	—	—	—	—	
−6	8.0	8.22	48.43	—	10.31	0.09	
151	Charge	6.3	46.0	43.8	10.2	—	400/400
−1	1.0	6.40	47.75	—	—	0.09	
−2	2.0	6.38	—	—	—	—	
−5	8.0	6.32	47.33	—	9.81	0.07	
187	Charge	16.0	60.0	30.0	10.0	—	400/400
−2	1.0	16.41	59.36	—	—	0.56	
−3	4.0	16.26	59.63	—	—	0.74	
−5	12.0	15.70	60.80	—	9.60*	0.60	
198	Charge	11.0	48.4	41.3	10.3	**	310/440
−1	1.0	10.88	47.13	—	—	1.49	
−4	8.0	10.83	47.83	—	10.40	1.70	
202	Charge	11.0	61.2	29.1	9.7	**	350/510
−1	1.0	11.04	60.75	—	—	0.15	
−4	8.0	11.07	60.14	—	9.53	0.10	
116	Charge	2.0	37.0	43.2	19.8	—	500/300
−1	1.0	2.19	36.47	—	—	0.14	
−3	5.0	2.19	37.30	—	—	—	
−5	7.0	2.24	37.00	—	20.70	0.04	
127	Charge	5.0	41.0	38.5	20.5	**	400/400
−2	1.0	5.19	40.98	—	—	0.62	
−3	2.0	—	40.93	—	—	—	
−5	7.0	5.18	41.23	—	20.43	—	
129	Charge	10.0	52.0	28.0	20.0	—	400/400
−1	1.0	10.23	51.69	—	—	0.11	
−3	5.0	—	51.98	—	—	—	
−5	8.0	10.27	51.87	—	20.78	0.12	
134	Charge	3.0	41.0	39.5	19.5	**	360/600
−1	1.0	3.00	40.73	—	—	0.03	
−2	2.0	3.04	—	—	—	—	
−5	8.0	3.13	40.43	—	19.23	—	
141	Charge	6.0	48.0	32.3	19.7	**	400/400
−1	1.0	6.05	47.60	—	—	0.10	
−2	2.3	6.10	—	—	—	—	
−5	8.0	6.10	47.90	—	21.05	0.12	
142	Charge	4.0	37.0	42.3	20.7	**	375/525
−1	0.5	3.69	37.41	—	—	0.09	
−2	1.0	3.67	—	—	—	—	
−5	8.0	3.58	37.75	—	20.61	0.12	
164	Charge	1.0	38.0	42.2	19.8	—	400/400
−2	0.7	1.23	38.03	—	—	0.60	
−3	4.0	1.30	37.23	—	—	0.02	
−5	8.0	1.40	36.75	—	24.02*	0.01	
166	Charge	7.0	51.9	28.3	19.8	**	350/730
−1	1.0	7.01	51.47	—	—	0.34	
−2	2.0	7.05	—	—	—	—	
−5	8.0	7.10	51.41	—	18.35*	0.10	
199	Charge	16.0	60.0	20.0	20.0	—	200/675
−1	1.0	15.80	62.31	—	—	0.26	
−4	8.0	15.73	61.28	—	18.16	0.54	

* Average of duplicate analyses.

** Used graphite crucible was either melted and cleaned or additions were made to residue. In either case, the new charge composition has been stated.

†† Nil: Denotes slags that were ignited to remove carbon prior to other chemical analyses. Carbon determinations are reported while a blank entry indicates that carbon was not determined.

Table II. Samples from Heats Made at 1500°C

Heat & Sample No.	Time at Temperature, Hr	Metal, Wt Pct Si	Slag Analyses, Wt Pct				Wt Slag/ Wt Metal (Grams)
			SiO_2	CaO	Al_2O_3	C	
83	Charge	7.8	40.0	50.0	10.0	**	300/300
—1	1.0	7.64	39.16	—	—	nil††	
—3	5.0	—	39.96	—	—	nil	
—5	8.0	7.48	40.08	49.29	9.90	nil	
88	Charge	3.8	37.0	53.0	10.0	**	300/300
—1	1.0	4.18	36.18	—	—	nil	
—3	5.0	4.16	—	—	—	—	
—5	8.0	4.32	36.02	51.67	10.82	nil	
101	Charge	10.3	43.5	46.6	9.9	—	300/500
—2	2.0	10.10	43.28	—	—	nil	
—3	5.0	9.83	—	—	—	—	
—5	8.0	10.03	43.56	46.95	9.92	nil	
113	Charge	6.0	45.5	44.8	9.7	—	400/400
—2	2.0	6.31	45.61	—	—	nil	
—3	5.0	6.44	—	—	—	—	
—8	8.0	6.45	44.92	—	—	nil	
—10	16.0	6.67	44.59	—	10.06	nil	
122	Charge	6.5	35.5	54.2	10.3	—	400/400
—1	1.0	6.66	36.32	—	—	nil	
—3	5.0	6.50	—	—	—	—	
—8	8.0	6.40	36.84	—	11.48	nil	
139	Charge	20.0	50.0	40.3	9.7	—	400/400
—1	1.0	20.15	48.66	—	—	—	
—3	3.0	20.20	—	—	—	—	
—8	8.0	20.08	49.92	—	10.54	—	
150	Charge	4.0	39.0	51.4	9.8	—	400/400
—1	0.5	4.00	40.51	—	—	0.33	
—2	1.0	4.01	40.90	—	—	0.27	
—6	7.8	4.09	41.28	—	10.14	0.21	
165	Charge	10.0	52.0	38.0	10.0	—	400/400
—2	0.5	10.04	50.77	—	—	0.06	
—5	4.0	10.33	—	—	—	—	
—6	8.0	10.55	49.66	—	11.00	0.06	
175	Charge	3.0	40.0	50.2	9.8	—	400/400
—2	1.0	3.20	39.67	—	—	0.08	
—3	3.0	3.20	—	—	—	—	
—5	7.5	3.29	39.63	—	9.52*	—	
176	Charge	6.0	38.0	51.8	10.2	—	400/400
—2	1.0	6.11	38.57	—	—	0.23	
—3	3.0	6.00	—	—	—	—	
—5	8.0	5.92	38.27	—	10.15*	0.11	
180	Charge	11.0	56.2	34.3	9.5	—	400/400
—1	1.0	11.10	56.31	—	—	0.10	
—3	4.0	11.35	—	—	—	—	
—5	12.0	11.83	55.72	—	10.05	0.07	
193	Charge	12.0	49.7	40.3	10.0	**	385/450
—1	1.3	12.12*	49.90*	—	—	0.04	
—4	8.0	12.42*	50.51*	—	9.48	0.04	
195	Charge	20.0	56.0	34.0	10.0	—	400/400
—1	1.0	20.02*	56.28	—	—	0.40	
—4	8.0	19.97*	55.71	—	10.01	0.32	
209	Charge	10.0	46.0	44.4	9.6	—	200/500
—1	0.5	10.36	44.90	—	—	0.16	
—4	8.0	10.22	45.57	—	9.29	0.16	
84	Charge	11.8	40.0	40.0	20.0	**	300/300
—2	3.0	11.98	40.56	—	—	nil	
—3	5.0	—	40.44	—	—	nil	
—5	8.0	11.70	40.52	39.59	19.99	nil	
90	Charge	6.3	36.0	44.0	20.0	—	400/250
—1	1.0	6.81	35.76	—	—	nil	
—2	3.0	6.73	35.54	—	—	nil	
—3	5.0	6.65*	—	—	—	—	
—5	8.0	6.77	35.95	43.21	19.73	nil	
100	Charge	18.0	49.0	31.5	19.5	—	400/500
—1	1.0	18.10	48.20	—	—	nil	
—3	5.0	17.93	—	—	—	—	
—5	8.0	17.70	48.42	32.45	19.64	nil	
114	Charge	10.5	42.0	38.4	19.6	—	400/400
—1	1.0	10.84	41.40	—	—	nil	
—3	5.0	10.80	—	—	—	—	
—5	8.0	10.75	41.42	—	—	nil	
—8	16.0	10.73	41.65	—	19.30	nil	
119	Charge	9.0	34.0	45.7	20.3	**	400/400
—2	2.0	9.28	34.77	—	—	nil	
—3	5.0	9.10	—	—	—	—	
—5	8.0	9.00	35.12	—	21.38	nil	
132	Charge	9.0	35.5	44.5	20.0	**	450/350
—1	1.0	9.10	35.68	—	—	—	
—3	5.0	9.02	—	—	—	—	
—5	8.0	8.96	36.19	—	21.58	—	
135	Charge	13.0	47.0	33.3	19.7	—	400/400
—1	1.0	13.63	44.72	—	—	0.42	
—2	2.0	13.52*	44.83	—	—	—	
—5	8.0	13.54*	44.93	—	20.23	0.24	
136	Charge	16.0	52.0	28.3	19.7	—	400/400
—1	1.0	15.74	51.30	—	—	0.30	
—3	2.0	15.94	—	—	—	—	
—4	7.0	15.94	51.50	—	19.79	0.42	
152	Charge	7.0	33.0	46.8	20.2	—	400/400
—2	1.0	6.85	33.83*	—	—	0.19	
—6	8.0	6.52	34.68	—	18.77	0.12	
162	Charge	17.4	51.5	28.7	19.8	**	380/600
—1	1.0	17.54	51.23	—	—	—	
—2	2.0	17.72	50.75	—	—	—	
—5	8.0	17.75	50.95	—	19.91	—	
173	Charge	3.0	44.0	38.0	18.0	—	150/600
—1	0.5	3.09	43.18	—	—	0.06	
—3	4.0	3.30	42.13	—	—	—	
—6	12.0	3.74	39.73*	—	19.56*	—	
197	Charge	21.5	50.0	29.8	20.2	—	200/600
—1	1.0	21.53	49.95	—	—	0.35	
—4	8.0	21.14	50.26	—	19.81	0.10	
200	Charge	3.8	37.0	43.2	19.8	**	140/580
—1	1.0	3.82	35.19	—	—	—	
—4	8.0	3.85	35.15	—	20.33	—	

* Average of duplicate analyses.

** Used graphite crucible was either melted and cleaned or additions were made to residue. In either case, the new charge composition has been stated.

†† Nil: Denotes slags that were ignited to remove carbon prior to other chemical analyses. Carbon determinations are reported while a blank entry indicates that carbon was not determined.

presence of SiO. The authors' results showed no pecularities below the silicon carbide solubility limit, and wherever complete analyses were made, the total based on normal valencies in the oxides was very close to 100 pct.

Humphrey, Todd, Coughlin, and King[4] have calculated for the reaction

$$SiO\ (g) + 2C\ (gr) = SiC\ (s) + CO\ (g) \quad [2]$$

a standard free-energy change at 1700°C of −16,700 cal. From this value it is found that the partial pressure of silicon monoxide in slags in equilibrium with silicon carbide, graphite, and 1 atm of carbon monoxide is 0.014 atm. In the authors' experiments, carbon monoxide flowed over the surface of the slag at a rate of about 150 ml per min. Assuming that the gas became saturated with silicon monoxide, the maximum amount that would be lost during an 8 hr run at 1700°C is 0.041 mol. In a typical slag weight of 200 grams containing 30 pct SiO_2, there is approximately one mol of silica. Thus, it is seen that the maximum evolution of silicon monoxide* can lead to a loss of about ½ of 1 pct of the amount of SiO_2 present per hour of operation. At lower temperatures the rate is much lower, and in all cases the effect on the accuracy of the results is insignificant.

* A similar calculation using experimental data on SiO pressures by H. Schäfer and R. Hörnle[5] indicates only about two thirds of the loss calculated above.

Accuracy of Analyses: Many duplicate analyses were obtained throughout the course of this investigation. Therefore, it is possible to calculate the mean

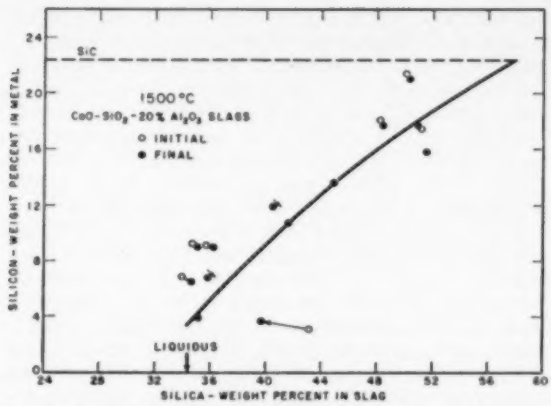


Fig. 4—Silicon distribution at 1500°C for Fe-Si-C alloys saturated with graphite and in equilibrium with CaO-SiO_2-20 pct Al_2O_3 slags under 1 atm of carbon monoxide.

Table III. Samples from New Heats Made at 1600°C

Heat & Sample No.	Time at Temperature, Hr	Metal, Wt Pct Si	Slag Analyses, Wt Pct SiO_2	CaO	Al_2O_3	C	Wt Slag/ Wt Metal (Grams)
172	Charge	15.0	44.0	38.0	18.0	—	150/600
−1	0.2	15.14	43.86	—	17.82	0.41	
−4	4.0	15.70	40.92	—	—	—	
−9	18.0	16.11	35.76	—	20.50	—	
179	Charge	15.0	29.0	50.0	21.0	—	150/600
−2	0.5	14.90	30.05	—	—	0.18	
−4	4.0	14.95	31.35	—	—	0.21	
−7	12.0	14.72	32.68	—	19.57	0.14	

difference of two analyses. Usually the duplicates were requested when the initial values reported were not consistent, e.g., when the silicon content of the metal and silica content of the slag both increased or decreased. Duplicates of slag analyses were divided about equally between samples suspected to be in error and samples on which duplication was initially requested, as, for instance, in master slags.

The statistical data on duplicability of analyses are shown in Table VI. The following definitions were used: x_i, the difference between individual pairs of analyses; $\bar{x}$, the arithmetic mean of the differences between pairs of analyses within a group; σ, the standard deviation from the mean difference; n, total number of pairs; and *range*, the highest and lowest values for the difference between pairs of analyses. The standard deviation was calculated by the formula

$$\sigma = \frac{\sqrt{\Sigma\,(x_i - \bar{x})^2}}{n}.$$

As an approximation, a normal distribution can be assumed, and the 95 pct confidence limits of two σ can be applied. On that basis, it may be concluded that in 95 pct of the times, duplicate silica analyses on the same sample will not vary by more than 0.25 + 2 (0.16), or 0.57 pct silica. On the other hand, this information may be applied to different silica samples in the same heat. Thus variations of 0.57 pct silica between two slag samples may be construed as a significant change in the melt composition 95 pct of the time. The last statement assumes no error due to sampling.

As pointed out previously,[2] it is unlikely that sampling errors are large compared to other factors that limit the accuracy of determination of the silicon-distribution curves. However, in order to eliminate

Table IV. Samples from Heats Made at 1700°C

Heat & Sample No.	Time at Temperature, Hr	Metal, Wt Pct Si	Slag Analyses, Wt Pct SiO_2	CaO	Al_2O_3	C	Wt Slag/ Wt Metal (Grams)
194	Charge	22.0	35.0	54.8	10.2	—	250/700
−1	1.0	21.63	36.13	—	—	0.40	
−3	4.0	21.45	37.15	—	9.63*	0.38	
201	Charge	23.0	37.0	52.7	10.3	—	200/500
−1	0.5	22.57	39.07	—	—	0.19	
−4	8.0	22.80	38.50	—	9.36	0.20	
203	Charge	19.0	36.0	53.7	10.3	—	200/500
−1	0.5	18.72	37.97	—	—	0.26	
−4	5.0	18.72	37.76	—	8.89	0.25	
204	Charge	15.0	37.8	53.2	9.0	**	175/600
−1	0.5	15.05	36.96	—	—	0.18	
−4	7.0	15.22	35.28	—	9.23	0.44	
205	Charge	18.0	38.5	52.1	9.5	**	175/600
−1	0.5	17.29	37.99	—	—	0.23	
−4	7.5	17.51	36.22	—	9.29	0.31	
206	Charge	22.0	41.0	49.5	9.5	—	200/500
−1	0.5	21.63	42.04	—	—	0.30	
−4	4.0	22.20	39.82	—	8.87	0.13	
207	Charge	19.0	39.8	51.2	9.0	**	175/600
−1	0.5	18.41	39.42	—	—	0.19	
−4	4.0	18.60	38.21	—	8.97	0.20	
158	Charge	21.4	32.0	48.0	20.0	—	300/700
−1	0.2	21.72	36.21	—	—	—	
−3	1.0	21.85	35.79	—	—	—	
−6	8.0	22.27	35.59*	—	20.83	—	
163	Charge	15.0	29.0	51.0	20.0	—	300/700
−1	0.5	14.97	28.49	—	—	0.28	
−2	1.0	14.95	28.49*	—	—	0.28	
−4	7.0	14.93	28.65	—	20.37	0.34	
167	Charge	20.0	28.7	50.9	20.4	**	250/750
−1	0.5	20.03	30.05	—	—	0.34	
−2	1.0	20.32	30.22	—	—	0.50	
−5	8.0	20.06	31.63	—	19.58	0.28	
168	Charge	19.0	34.6	44.6	20.8	**	250/760
−1	0.5	18.85	33.95	—	—	0.29	
−2	1.0	18.85	33.73	—	—	0.21	
−5	8.0	18.91	31.22*	—	23.32	0.20	
169	Charge	10.0	28.0	52.3	19.7	—	300/700
−1	0.5	10.14	28.14	—	—	0.28	
−2	1.0	10.21	27.74	—	—	0.31	
−5	8.0	10.55	26.41	—	19.25	0.62	
170	Charge	15.0	44.0	38.0	18.0	—	150/600
−1	0.1	15.38	43.53	—	—	0.17	
−3	1.0	16.08	39.03	—	—	0.08	
−5	7.0	16.96	30.78	—	21.93	—	
184	Charge	20.1	31.6	48.4	20.0	**	220/680
−2	1.0	19.95	29.29	—	—	0.89	
−3	3.0	19.93	29.50	—	—	0.68	
−5	8.0	19.65	30.84	—	20.43*	0.76	
185	Charge	22.2	33.5	46.5	20.0	**	250/800
−2	1.0	22.68	32.11	—	—	0.24	
−3	3.0	22.46	32.16	—	—	0.23	
−5	7.8	22.40	32.48	—	20.25	0.20	
208	Charge	18.0	28.0	51.8	20.2	—	200/500
−1	0.2	18.38	28.60	—	—	0.92	
−4	4.0	17.87	30.59	—	18.79	0.54	
210	Charge	20.0	34.0	46.2	19.8	—	200/500
−1	0.5	20.04	34.14	—	—	0.29	
−4	4.0	20.06	33.72	—	19.28	0.18	

* Average of duplicate analyses.
** Used graphite crucible was either melted and cleaned or additions were made to residue. In either case, the new charge composition has been stated.

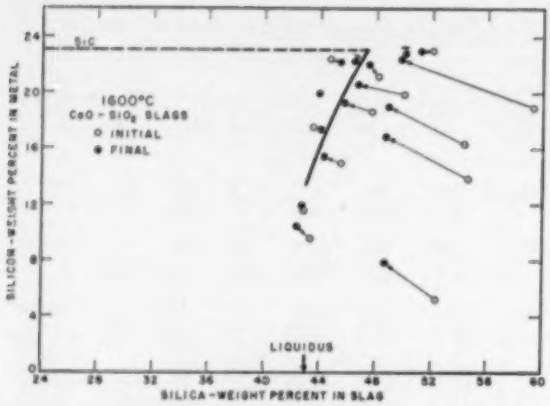


Fig. 5—Silicon distribution at 1600°C for Fe-Si-C alloys saturated with graphite and in equilibrium with CaO-SiO_2 slags under 1 atm of carbon monoxide.

anomalies in composition changes, the first sample analyzed has been plotted in Figs. 1 to 10, rather than the calculated charge. Similarly, the last analysis has been plotted with a correction for carbon but not for alumina. The final alumina content was accounted for when the equilibrium curves were drawn.

Discussion of Results

Slowness of Reaction and Its Influence on Accuracy: It is well known that silica reduction (or the reverse reaction) is a slow process below 1600°C. This was especially evident from the work of Grant, Troili, and Chipman[1] who studied the effect of silica reduction on desulphurization at 1525°C. They found that nine hours was insufficient time for equilibration when the metal phase was required to change from 4 to 1 pct or from 0 to 1 pct Si.

Referring to Figs. 1 to 10, it can be seen how their conclusion has been confirmed at 1425° and 1500°C. At 1425°C (Figs. 1 and 2), very little reaction occurred in the eight hours allowed for most heats. At 1700°C (Figs. 9 and 10), a significant amount of reaction occurred in four hours, even when the initial composition was not displaced far from equilibrium.

The very slow nature of the reaction has been the major factor in limiting the accuracy with which the equilibrium curves could be determined at 1425°, 1500°, and 1600°C. At 1700°C the reaction rate increased to the extent that there was rather good convergence on the equilibrium distribution; however, the experiments covered such a small concentration interval that the shape of the curve was not well defined. In view of all the factors involved, it seems reasonable to place the limits of error at ±1 pct silica at a given silicon level. At the high silica end of Figs. 1 and 2 at 1425°C, the experimental error is substantially larger. Fortunately, however, there is a theoretical method (discussed in a later section), by means of which the upper extremities of these lines may be fixed with about as good accuracy as at other temperatures.

The slowness of the reduction process which contributed so much to the difficulty of the experiments also must be credited with contributing to the success of the blast-furnace process. If the hot metal actually reached equilibrium with blast-furnace slag, practically all of it would require desiliconizing before use.

Range of Silica Contents Studied: In general, the distribution of silicon between slag and metal has been extended over the widest possible range at the temperatures and alumina concentrations employed. The relations between alumina content, the temperature, and the phase which precipitates at the upper and lower silica compositions have been summarized in Table VII. The silica concentrations corresponding to these limits may be read from the lime-alumina-silica phase diagram.[8] In general, the lower limits of the authors' results agree well with the diagram and the solubility limits shown in

Table V. Samples from Slags Containing MgO at 1600°C

Heat & Sample No.	Time at Temperature, Hr	Metal, Wt Pct Si	Slag Analyses, Wt Pct					Wt Slag/ Wt Metal (Grams)
			SiO_2	CaO	Al_2O_3	MgO	C	
147	Charge	4.5	36.8	43.2	10.0	10.0	—	300/700
—1	0.5	4.55	35.50	—	—	—	0.22	
—2	1.0	4.66	35.53	—	—	—	0.22	
—6	8.0	4.94	34.31*	46.09*	9.61*	9.54*	0.13	
155	Charge	21.0	44.0	36.0	10.0	10.0	—	300/700
—1	0.5	19.91	44.25	—	—	—	0.43	
—2	1.0	20.31	44.04	—	—	—	0.28	
—6	8.0	20.47	44.25	35.40*	10.09*	10.10*	—	
156	Charge	15.0	40.0	40.0	10.0	10.0	—	300/700
—1	0.5	14.81*	40.64	—	—	—	0.10	
—2	1.0	14.21*	40.32	—	—	—	—	
—6	8.0	14.88*	40.53*	39.79	9.51	9.71	0.13	
178	Charge	18.0	40.5	39.5	10.0	10.0	**	280/730
—2	1.0	18.20*	38.11*	—	—	—	0.39	
—5	8.0	17.89	40.86	—	10.49	10.02	0.61	
181	Charge	12.0	36.0	44.0	10.0	10.0	—	300/700
—2	1.0	11.79	35.81	—	—	—	0.17	
—3	3.0	11.75*	—	—	—	—	—	
—5	8.0	11.92	35.62	—	9.76	9.43	0.28	
190	Charge	11.9	33.5	46.5	10.0	10.0	**	280/670
—1	1.0	11.86	33.35	—	—	—	0.43	
—3	6.0	11.59	34.82	45.90	10.11*	9.53	0.39	

* Average of duplicate analyses.

** Used graphite crucible was either melted and cleaned or additions were made to residue. In either case, the new charge composition has been stated.

Table VI. The Mean Difference of Two Analyses

Variable, Wt Pct	Number of Pairs, n	Mean Difference, $\bar{x}$	σ	Range
SiO_2	94	0.25	0.16	0.00 to 0.77
CaO	35	0.27	0.18	0.00 to 0.56
10 pct Al_2O_3	19	0.18	0.14	0.01 to 0.50
20 pct Al_2O_3	45	0.51	0.34	0.03 to 1.32
0 to 8 pct Si	6	0.02	0.02	0.00 to 0.04
8 to 15 pct Si	13	0.10	0.08	0.00 to 0.23
15 to 24 pct Si	28	0.23	0.19	0.04 to 0.62

Table VII and in Figs. 1 through 10 are averages between the two. At temperatures above 1450°C, the upper limit of SiO_2 is set by the occurrence of silicon carbide as a stable phase. This is discussed in the following section and the observed upper limits are shown in Table VII.

Stability of Silicon Carbide: The occurrence of cubic silicon carbide at 1600°C was discussed in the original report.[2] The silicon concentration of the metal at which silicon carbide becomes stable in the temperature range of 1200° to 1700°C has now been established more accurately[7] and the data are summarized in Table VIII.

In their study of the electrolysis of CaO-SiO_2 slags, Bockris, Kitchener, and Davies[8] found that silicon carbide was stable at 1600°C in slags containing 50 mol pct (51.7 wt pct) silica. The authors' data in Fig. 7 indicate that the limit of stability is about 48 wt pct. This is the only series that actually was carried to high enough silica levels to form SiC.

Taylor and Laidler[9] studied the formation of silicon carbide by the reaction

$$SiO_2 \text{ (crist.)} + 3C \text{ (graphite)} = SiC\ (\beta) + 2CO\ (g). \quad [3]$$

The minimum temperature at which they were able to form silicon carbide was 1450°C. This may be taken as experimental evidence that 1450°C is the temperature at which all substances in Eq. 3 can co-exist at unit activity. Above 1450°C silica must be at less than unit activity, and below this temperature silicon carbide does not form in the slag.

The free energy of formation of both SiO_2 and SiC are now known accurately. The former depends principally upon the heat of formation of cristobalite recently determined by Humphrey and King[10] and thermal data which have been assembled by Kelley.[11] The equation which results from these data and which already has been used in establishing the activity of silicon[7] is

$$Si\ (1) + O_2\ (g) = SiO_2\ (\text{crist.}) \qquad \Delta F^\circ = -217{,}700 + 47.0T. \quad [4]$$

The heat of formation of β silicon carbide and its heat capacity at high temperatures were determined by Humphrey et al.[4] Their data lead to a heat of formation of SiC from liquid silicon and graphite at about 1700°K of -24.4 ± 0.9 kcal. A slight adjustment within the accuracy of the data along with Kelley's tables[11] yields the following equation which is in exact agreement with activity data on SiC-saturated solutions[7]

$$Si\ (1) + C\ (\text{graphite}) = SiC\ (\beta) \qquad \Delta F^\circ = -24{,}700 + 8.3T. \quad [5]$$

For the free energy of carbon monoxide, an equation is quoted from *Basic Open Hearth Steelmaking* which was derived from the National Bureau of Standards selected values:

$$2C\ (\text{graphite}) + O_2\ (g) = 2CO\ (g) \qquad \Delta F^\circ = -56{,}200 - 40.4T. \quad [6]$$

The above equations may be combined to determine the free-energy change in Eq. 3:

$$SiO_2\ (\text{crist.}) + 3C\ (\text{graphite}) = SiC\ (\beta) + 2CO\ (g) \qquad \Delta F^\circ = 136{,}800 - 79.1T. \quad [7]$$

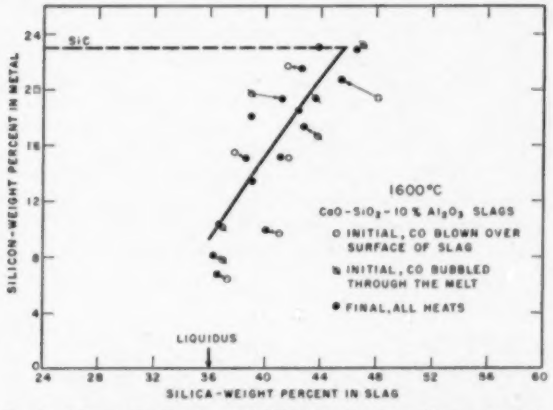

SiC
1600°C
CaO - SiO2 - 10 % Al2O3 SLAGS
INITIAL, CO BLOWN OVER SURFACE OF SLAG
INITIAL, CO BUBBLED THROUGH THE MELT
FINAL, ALL HEATS
LIQUIDUS
SILICON - WEIGHT PERCENT IN METAL
SILICA - WEIGHT PERCENT IN SLAG

Fig. 6—Silicon distribution at 1600°C for Fe-Si-C alloys saturated with graphite and in equilibrium with CaO-SiO_2-10 pct Al_2O_3 slags under 1 atm of carbon monoxide.

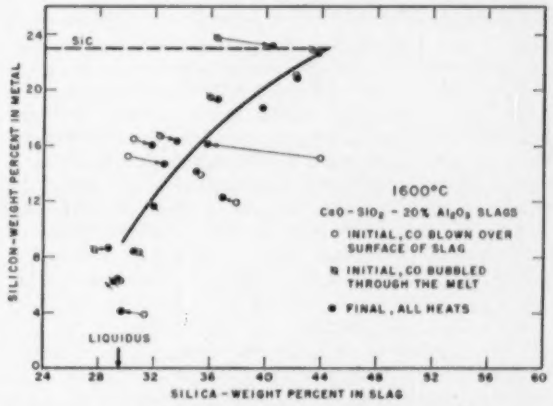

SiC
1600°C
CaO - SiO2 - 20% Al2O3 SLAGS
INITIAL, CO BLOWN OVER SURFACE OF SLAG
INITIAL, CO BUBBLED THROUGH THE MELT
FINAL, ALL HEATS
LIQUIDUS
SILICON - WEIGHT PERCENT IN METAL
SILICA - WEIGHT PERCENT IN SLAG

Fig. 7—Silicon distribution at 1600°C for Fe-Si-C alloys saturated with graphite and in equilibrium with CaO-SiO_2-20 pct Al_2O_3 slags under 1 atm of carbon monoxide.

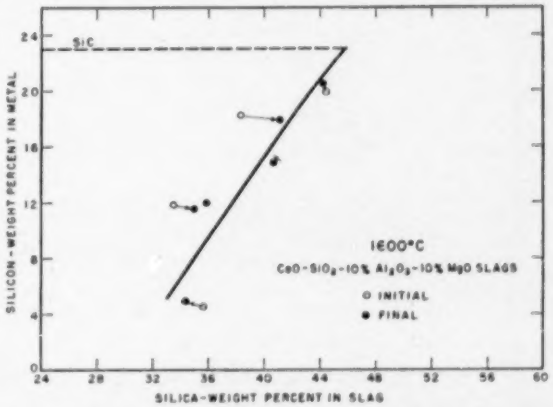


Fig. 8—Silicon distribution of Fe-Si-C alloys saturated with graphite and in equilibrium with CaO-SiO_2-10 pct Al_2O_3-10 pct MgO under 1 atm of carbon monoxide.

Table VII. Limiting Slag Compositions

Temperature, °C	Al_2O_3, Pct	Lower Limit		Upper Limit		Upper Limit in Graphite	
		SiO_2, Pct	Phase	SiO_2, Pct	Phase	SiO_2, Pct	Phase
1425	10	39.2	Ca_2SiO_4	69	Tridymite	69	Tridymite
	20	37.0	Gehlenite	71	Mullite	71	Mullite
1500	10	37.0	Ca_2SiO_4	76	Cristobalite	57.5	SiC
	20	34.4	Gehlenite	72	Mullite	58.0	SiC
1600	0	42.8	Ca_2SiO_4	68	Cristobalite	46.5	SiC
	10	36.4	Ca_2SiO_4	—	none	45.8	SiC
	20	29.2	Ca_2SiO_4	74.5	Mullite	44.2	SiC
1700	10	35.5	Ca_2SiO_4	—	none	38.5	SiC
	20	27.0	Ca_2SiO_4	78	Mullite	35.0	SiC

From this free-energy relation, the activity of silica can be calculated for slags in equilibrium with pure graphite, β silicon carbide, and 1 atm of carbon monoxide. In this way values for the activity of silica at several temperatures have been calculated and are shown in Table VIII. The activity refers to a standard state of β cristobalite. The ratio of the activity of silicon in the metal to that of silica in the slag is also tabulated in the table. This ratio, which is the equilibrium constant of Eq. 1, is independent of the presence of SiC. It will be used in a later section to calculate the activity of silica from the known activity of silicon in the equilibrium metal.

It is also shown in Table VIII that 1456°C is the temperature at which all substances in Eq. 3 can co-exist in their standard states. This is very good agreement with the experimental value of 1450°C found by Taylor and Laidler.[9]

Equilibrium at 1425°C: At 1425°C the more acid slags were too viscous to be studied up to the liquidus compositions, but some information on how to draw the curves of Figs. 1 and 2 can be obtained from the above discussion. It is evident that at 1425°C, the 10 pct alumina slags can be studied up to silica saturation without formation of silicon carbide. The solubility of cristobalite is 69 pct SiO_2 and at this composition $a_{SiO_2} = 1$. From the data of Table VIII it is found that in the metal phase $a_{Si} = 0.20$ which corresponds to $N_{Si} = 0.312$ or a concentration of 18.8 pct Si. The upper limit of the line in Fig. 1 is taken therefore as 18.8 pct Si at 69 pct SiO_2.

An analogous situation occurs in Fig. 2 for the 20 pct alumina slags. In this case, mullite ($3Al_2O_3 \cdot 2SiO_2$) precipitates. Since it is obvious that the silica activity in mullite is less than that in pure silica, the curve has been drawn so as to intersect its liquidus at a lower silicon concentration than in Fig. 1. This concentration is fixed approximately by the following considerations: In this region of the phase diagram, it is evident from the shapes of the silica solubility isotherms that the silica activity in a 20 pct alumina slag is equal to that of about 5 pct lower silica concentration in the 10 pct alumina series. Hence the silicon concentration at 71 pct SiO_2 in Fig. 2 is about equal to that at 66 pct SiO_2 in Fig. 1, or 17 pct Si. This is plotted as the upper extremity of the line in Fig. 2.

Effect of MgO: Fig. 8 contains the data on slags in which 10 pct Al_2O_3 and 10 pct MgO were present at 1600°C. The equilibrium curve in this figure is identical with that of Fig. 6 in which 10 pct Al_2O_3 and no MgO were present at the same temperature. The agreement indicates that 10 pct MgO has the same effect as an equal weight per cent of CaO except that the range of fluid slags is extended to lower percentages of silicon. The equivalence of MgO and CaO on a weight basis rather than in terms of mol fractions has been observed in the same range of compositions in studies of the desulphurizing powers of similar slags.[10]

Summary—Effects of Temperature and Alumina: As a means of illustrating the profound effect of temperature and composition on the equilibrium curves, the data from Figs. 1 to 10 have been summarized in Fig. 11. It might be pointed out again that the interest in silicon distribution between blast-furnace slag and metal stems from desulphurization studies. In view of the practical nature of the results, it was considered useful to express the data in weight percent. Translation to mol fractions will be discussed later.

Referring to Fig. 11, it will be seen that the data are grouped according to temperature. With increasing temperature, there has been a shift to lower silica concentrations and an increase in the slope of the curves.

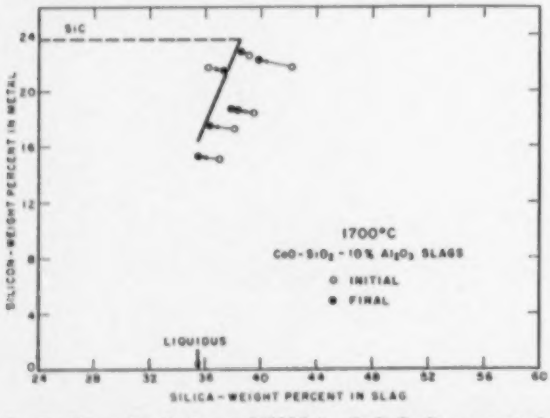


Fig. 9—Silicon distribution at 1700°C for Fe-Si-C alloys saturated with graphite and in equilibrium with CaO-SiO_2-10 pct Al_2O_3 slags under 1 atm of carbon monoxide.

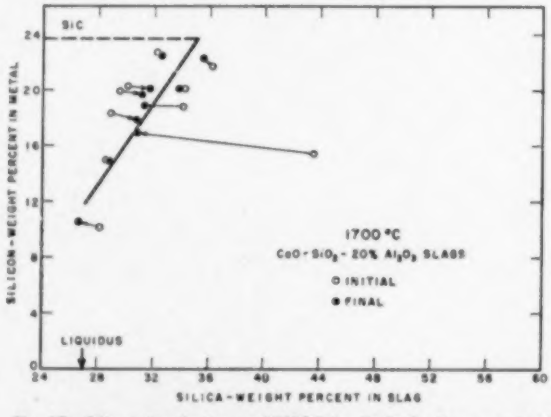


Fig. 10—Silicon distribution at 1700°C for Fe-Si-C alloys saturated with graphite and in equilibrium with CaO-SiO_2-20 pct Al_2O_3 slags under 1 atm of carbon monoxide.

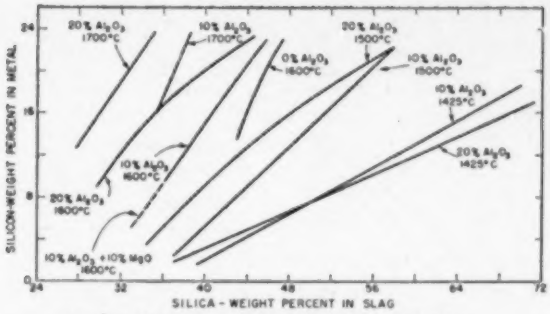


Fig. 11—Summary of silicon-reduction equilibria.

The effect of the substitution of 10 wt pct alumina for 10 wt pct lime has been studied at all temperatures. At 1500°, 1600°, and 1700°C, increasing the alumina has caused an increase in the silicon content. The effect is greater in the more basic slags. At 1425°C, on the other hand, it is only in the more basic slags that this effect persists. At the acid end of the range, substitution of alumina for lime decreases the silicon content of the metal.

Influence of Sulphur: It has been pointed out that silicon-equilibrium data are needed for subsequent work on the rate of sulphur removal from liquid pig iron. Therefore, it is worthwhile to compare the silicon equilibrium obtained with and without sulphur present.

Fig. 12 shows silicon distribution in the experiments of Hatch and Chipman[12] and of Filer and Darken[13] at 1500°C with 1.5 to 2.0 pct S in the final slag. The changes in metal and slag compositions during the run are indicated where known; otherwise, the direction of change in silicon content is shown by an arrow. The small numeral beside each point denotes the percentage of Al_2O_3 in the final slag. The magnesia content was generally between 5 and 15 pct, but it may be assumed that this is unimportant to the present discussion, since its only evident effect was in lowering the silica limit. Similar data of Hatch and Chipman at higher sulphur contents are shown in Fig. 13. The lines representing sulphur-free slags of 10 to 20 pct Al_2O_3 are shown for comparison.

Inspection of these figures shows that sulphur has a tendency to increase the silicon content of the metal. Practically all heats have finished at a higher silicon content than that which would be predicted by extrapolation of a line for slags of corresponding alumina content. This is not a very satisfactory comparison, however, since there are no grounds for belief that the lines can be extrapolated linearly. In slags of higher sulphur content, Fig. 13, the effect is more marked. Consider, for example, two slags containing 34 pct SiO_2 and 12 pct Al_2O_3 with metal samples of 2.1 to 2.5 pct Si. In the absence of sulphur, such slags would have contained about 37 pct

Table VIII. Compositions and Activities in Equilibrium with Both Graphite and SiC

Temperature, °C	Si, Pct	C, Pct	N_{Si}	a_{Si}	a_{SiO_2}	a_{Si}/a_{SiO_2}
1425	21.9	0.28	0.355	0.043	2.1	0.0205
1456	—	—	—	—	1.0	—
1500	22.4	0.33	0.362	0.059	0.38	0.155
1600	23.0	0.41	0.369	0.085	0.048	1.77
1700	23.7	0.50	0.376	0.120	0.0074	16.2

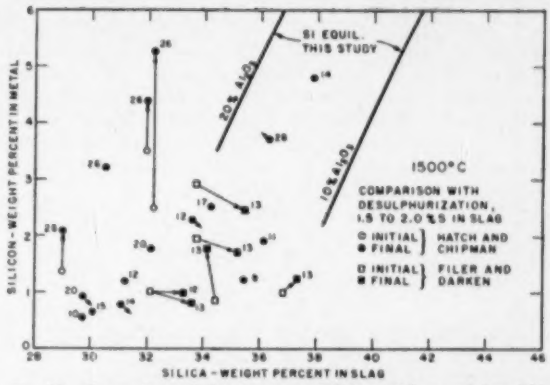


Fig. 12—Comparison of the silicon equilibrium without sulphur (this study) and the silicon equilibrium obtained in the presence of sulphur at 1500°C. Numbers beside the individual points indicate alumina.

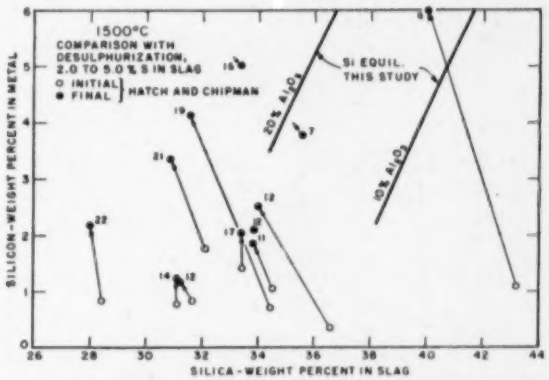


Fig. 13—Comparison of the silicon equilibrium without sulphur (this study) and the silicon equilibrium obtained in the presence of sulphur at 1500°C. Numbers beside individual points indicate alumina.

SiO_2 for the same metal silicon. Similarly, slags of 19 to 21 pct Al_2O_3 and 3.4 to 4.2 pct Si, if they had contained no sulphur, would have required about 3 pct higher SiO_2. Since the sulphur content of the metal is not high, it is evident that the shift in silicon distribution must be ascribed to the fact that sulphur increases the activity coefficient of silica in the slag.

In the corresponding experiments at 1425°C, the approach to silicon equilibrium was so slow that any effect of sulphur necessarily was masked. In the work of Grant, Kalling, and Chipman,[14] silicon equilibrium was not approached because of the oxidizing effect of MnO. Grant, Troili, and Chipman[3] approached silicon equilibrium more closely and their results, shown in Fig. 14, confirm the conclusion that sulphur increases the activity coefficient of silica.

The effect of sulphur is probably a simple replacement of oxide ion by sulphide ion or, in other words, the partial neutralization of lime. The effect cannot be established quantitatively from the data cited, but it appears to be approximately what would be expected on the basis of neutralization of 1 mol of CaO by each atom of sulphur.

Activities in the Slags

Silica: The activity of silica in the slag is related directly to that of silicon in the equilibrium metal through the known free-energy change of Eq. 1 which is obtained from Eqs. 4 and 6 and is rewritten

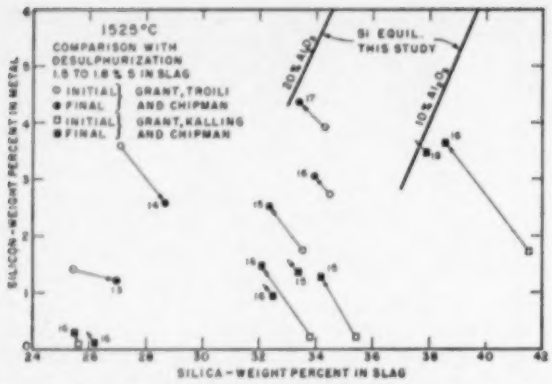


Fig. 14—Comparison of the silicon equilibrium obtained in the presence of sulphur at 1525°C. Numbers beside individual points indicate alumina.

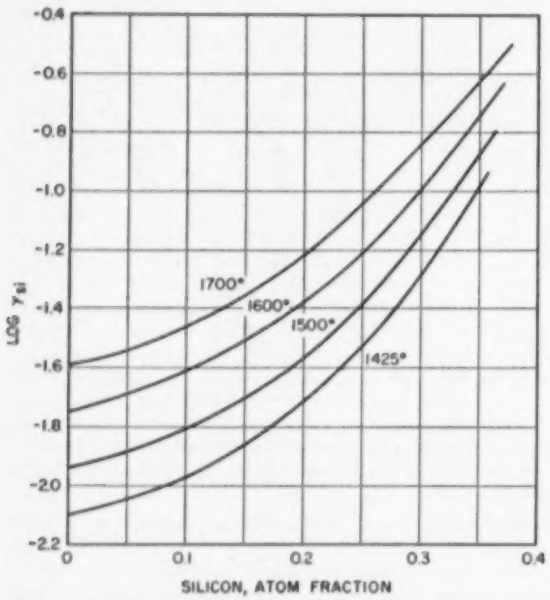


Fig. 15—Activity coefficient of Si in graphite-saturated Fe-C-Si solutions.

$$SiO_2\,(crist.) + 2C\,(graphite) = Si\,(1) + 2CO\,(g)$$
$$\Delta F^\circ = +161{,}500 - 87.4T. \quad [8]$$

The ratio of the activity of silica to that of silicon is given in Table VIII. The activity coefficient of silicon in graphite-saturated iron has been determined by Chipman, Fulton, Gokcen, and Caskey,[7] whose data are reproduced in Fig. 15. The atom fraction and activity coefficient of Si determine its activity which, by means of the ratio given in the last column of Table VIII, establishes the activity of SiO_2 in the equilibrium slag. Thus every point on each line of Fig. 11 yields a value for the activity of SiO_2 at a given temperature and composition. The data for the slags at 1600°C are shown in Fig. 16 in which the logarithm of the activity of silica is plotted against mol fraction of silica. In converting weight percent to mol fraction in the slag, it was noted that, throughout the composition range of the ternary slags considered, 10 and 20 wt pct Al_2O_3 correspond respectively to 6.0 and 12.5 mol pct. In the quaternary series, 10 pct each of Al_2O_3 and MgO correspond to 5.7 and 14.4 mol pct, respectively.

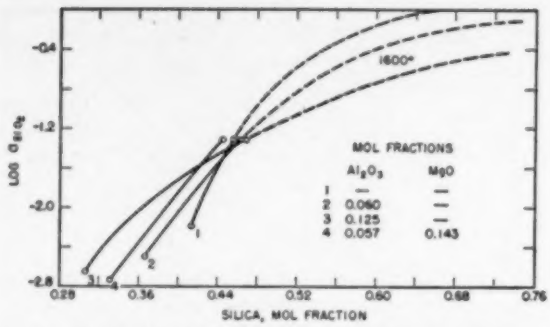


Fig. 16—Activity of SiO_2 (referred to cristobalite) at 1600°C.

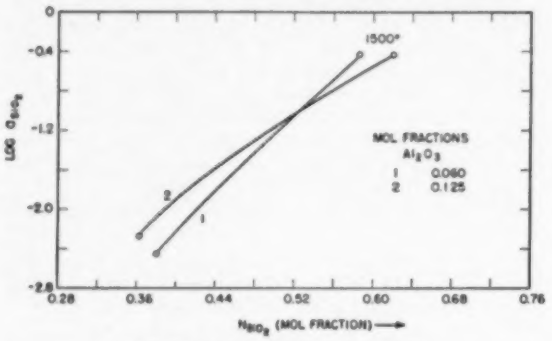


Fig. 17—Activity of SiO_2 at 1500°C.

The composition ranges covered by experimental measurement are denoted by solid lines in Fig. 16. The broken lines representing extrapolations are based on the following considerations: In the binary system, the solubility of cristobalite is 68 pct or $N_{SiO_2} = 0.664$. At this composition log $a_{SiO_2} = 0$ and the experimental line is extrapolated to this point. In the 10 pct alumina series, the activity of silica is always less than one. At $N_{Si} = 74$ in this series, it is about equal to that at $N_{Si} = 61$ in the binary system, as judged by isothermals of the phase diagram; and the extrapolation is made accordingly. Similar extrapolation of the 20 pct alumina series is fraught with greater uncertainty. The three curves cross at about $N_{SiO_2} = 0.45$. At higher silica, substitution of Al_2O_3 for CaO on a molar basis decreases the activity of silica.

In comparing the series containing alumina and magnesia with the other data, it is now clear that substitution of MgO for CaO on a molar basis increases the activity of SiO_2. This conclusion must be restricted to the experimental range of compositions.

Activity of silica at the other temperatures is shown in Figs. 17 and 18. It must be emphasized that the shapes of these curves are not well determined by the experimental data. Nevertheless certain phenomena are common to all series. In general the curves are steeper at lower concentrations. The reversal in the effect of alumina is observed at the three lowest temperatures, and the 1700°C curves show the same tendency.

The effect of temperature on the activity coefficient of silica is remarkably small, a fact brought out clearly in Fig. 18 where the 1700°C lines appear almost as extensions of those for 1425°C. The significance is that the heat of solution of cristobalite in these slags is small.

The relation of the experimental data at 1600°C to the ternary phase diagram is shown in Fig. 19.

Table IX. Aluminum in Metal at 1600°C

Heat and Sample No.	Al, Pct	Heat and Sample No.	Al, Pct
49-6	0.018	92-5	0.024
117-5	0.013	54-6	0.023
118-5	0.015	66-6	0.013
125-4	0.021	87-4	0.036
97-5	0.010	103-5	0.025
52-5	0.020	107-5	0.026
76-4	0.028	109-4	0.016
86-5	0.031	124-5	0.025

It is seen readily that the data cover only a small part of the entire liquid region, although this is the part of greatest interest for blast-furnace slags. Compositions above the 0.05 line react with graphite to form silicon carbide; in this region, the lines represent estimates.

Activity of Lime: For slags in equilibrium with dicalcium silicate, the activities of CaO and SiO_2 are related, at a given temperature, through the equation $a^2_{CaO} \cdot a_{SiO_2}$ = constant. The numerical value of the constant is obtainable from the free energy.

The heat of formation of calcium orthosilicate has been redetermined by King.[12] For the formation of β Ca_2SiO_4 from CaO and quartz at 25°C, he finds that $\Delta H = -30{,}190 \pm 230$ cal. This agrees well with the −29,800 value deduced by Richardson, Jeffes, and Withers[16] from related data. Recent measurements of Todd[17] yield an entropy change of +1.5 units at 298°K for formation of the silicate from the oxides. With the aid of Kelley's tables,[11] these data yield the following equation at 1600°K, which may be taken as a good approximation over the authors' experimental-temperature range:

$$2CaO\ (s) + SiO_2\ (s) = Ca_2SiO_4\ (\beta)$$
$$\Delta F^\circ = -26{,}200 - 4.9T. \quad [9]$$

From this equation the free-energy change at 1873°K is −35,400 cal and the activity product is 7.4×10^{-6}. For slags in equilibrium with Ca_2SiO_4 at 1600°C and containing 0, 10, and 20 pct Al_2O_3, this leads to values of a_{CaO} of 0.11, 0.15, and 0.18. Here the standard state is the pure solid CaO. The calculations depend rather heavily upon measurements of the heat capacities of reactants and product at high temperatures. Data quoted by Kelley indicate a value for $\Delta^\circ C_p$ of about 5 cal per degree, a remarkably large value. If the usual estimate of $\Delta^\circ C_p = 0$ is applied, as recommended by Richardson, Jeffes, and Withers,[16] the free-energy change is −33,000 cal and a_{CaO} is increased by 50 pct.

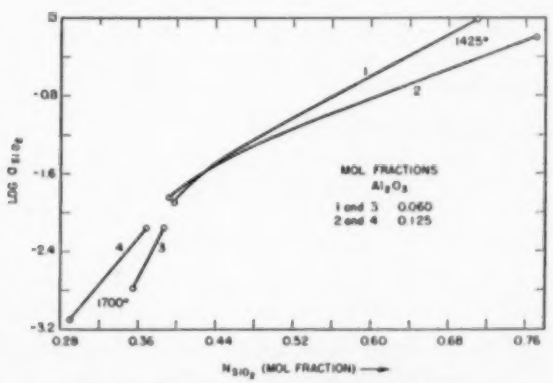


Fig. 18—Activity of SiO_2 at 1425° and 1700°C.

Activities of silica and lime in the binary solution are shown in Fig. 20. The activities of CaO were obtained from those of SiO_2 through the Gibbs-Duhem equation beginning with the value mentioned above for a slag saturated with orthosilicate. The results constitute a remarkable experimental confirmation of calculations by Richardson[16] based on the phase diagram and free energies of the calcium silicates. Activities of CaO and Al_2O_3 in the ternary system could be calculated from that of SiO_2 if the latter were available for a wide range of compositions. The authors' data seem inadequate for this purpose and the computation will be deferred until more data are available.

Alumina: A number of metal samples from the 1600°C heats were analyzed for aluminum in the hope that the results might establish the activity of alumina. Since there are no comparable data in the literature, the results are recorded for possible future use in Table IX. At the present time, the effects of silicon and carbon on the activity coefficient of aluminum are unknown; and since these may be rather large, any attempt to calculate the activity of alumina from the data would be premature.

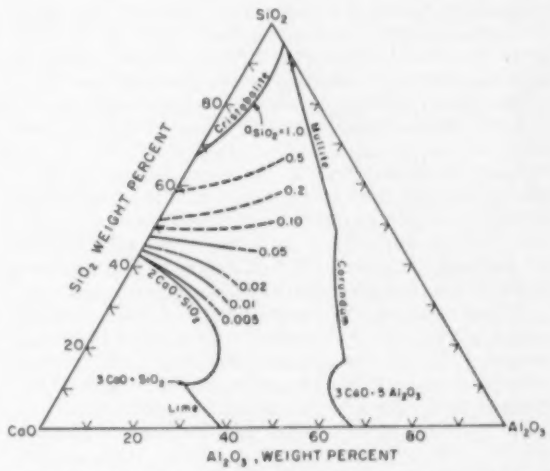


Fig. 19—CaO-Al_2O_3-SiO_2 at 1600°C showing lines of constant silica activity.

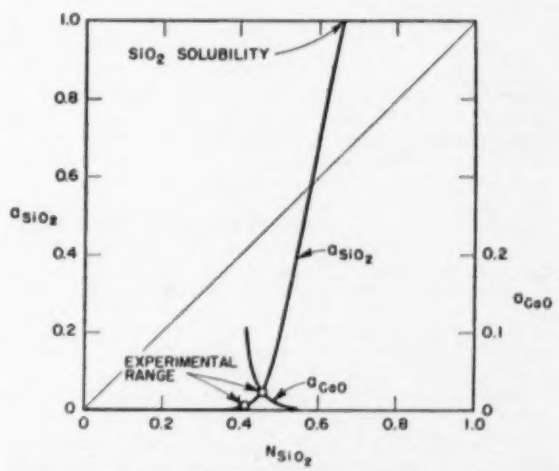


Fig. 20—Activities of SiO_2 and CaO in their binary solution at 1600°C.

Summary

Experimental data are presented on the reduction of silicon from blast-furnace-type slags by carbon-saturated iron. The reaction is very slow, especially at the lowest temperature studied, 1425°C. At 1600°C under conditions of rapid stirring, the approach to equilibrium often required more than eight hours. Even at 1700°C the rate is rather slow. This slowness adds to the experimental difficulties but is a blessing to blast-furnace operation. By studying many compositions and approaching equilibrium from both sides in an atmosphere of carbon monoxide, curves were obtained showing the equilibrium silicon content of Fe-C-Si alloys as a function of temperature and slag composition.

An upper limit to the silica content of slags in equilibrium with graphite is determined by the appearance of β silicon carbide as a stable phase, and the conditions for its occurrence have been established.

In the more basic slags, the replacement of lime by an equal weight of alumina increases the equilibrium concentration of silicon in the metal. In very acid slags, the reverse effect is found. Replacement of lime by an equal weight of magnesia does not change the silicon distribution but extends the range of fluid slags to lower silica contents. Comparison with results of desulphurization studies indicates that sulphur in the slag increases the silicon content of the metal. This appears to be due to neutralization of CaO, or replacement of oxide ion by sulphide ion.

The activity of silica in the slags has been determined from the silicon content of the equilibrium metal, its known activity coefficient, and available free-energy data. The activity coefficient of silica in the slag, referred to cristobalite as the standard state, is not greatly affected by temperature.

The activity of lime was calculated from the free energy of Ca_2SiO_4 and the silica activity of slags saturated with this compound. Activities of CaO between 0.1 and 0.2 were found on the acid side of this composition. Results are shown graphically.

Aluminum content of the equilibrium metal is reported, but alumina activities in the slags remain unknown.

Acknowledgments

This work is a part of a series of studies of the chemical reactions of blast-furnace slag and metal sponsored by the American Iron and Steel Institute. The authors express their thanks to Professor N. J. Grant and Messrs. J. C. D'Entremont, Henry Kaplan, Warren Hammond, and Richard Robichaud. They acknowledge with thanks the numerous and careful chemical analyses carried out by Mr. D. L. Guernsey and his staff.

References

[1] N. J. Grant, O. Troili, and J. Chipman: *Trans.* AIME (1951) **191**, p. 672; JOURNAL OF METALS (August 1951).

[2] J. C. Fulton, N. J. Grant, and J. Chipman: *Trans.* AIME (1953) **197**, p. 185; JOURNAL OF METALS (February 1953).

[3] P. V. Gel'd and O. A. Esin: *Doklady Akademii Nauk SSSR* (1950) **70**, p. 473.

[4] G. L. Humphrey, S. S. Todd, J. P. Coughlin, and E. G. King: United States Bureau of Mines, Report of Investigation 4888 (1952).

[5] H. Schäfer and R. Hörnle: *Ztsch. anorg. allgem. Chemie* (1950) **263**, p. 261.

[6] *Phase Diagrams for Ceramists.* Edited by F. P. Hall and H. Insley. (1947) p. 67. Am. Ceramic Soc.

[7] J. Chipman, J. C. Fulton, N. Gokcen, and G. R. Caskey: *Acta Metallurgica* (1954) **2**, p. 439.

[8] J. O'M. Bockris, J. A. Kitchener, and A. E. Davies: *Trans.* Faraday Soc. (1952) **48**, p. 536.

[9] A. Taylor and D. S. Laidler: *British Journal of Applied Physics* (1950) **1**, p. 174.

[10] G. L. Humphrey and E. G. King: *Journal* ACS (1952) **74**, p. 2041.

[11] K. K. Kelley: United States Bureau of Mines Bulletins 476 (1949), 477 (1950).

[12] G. G. Hatch and J. Chipman: *Trans.* AIME (1949) **185**, p. 274; JOURNAL OF METALS (March 1949).

[13] E. W. Filer and L. S. Darken: *Trans.* AIME (1952) **194**, p. 253; JOURNAL OF METALS (March 1952).

[14] N. J. Grant, U. Kalling, and J. Chipman: *Trans.* AIME (1951) **191**, p. 666; JOURNAL OF METALS (August 1951).

[15] E. G. King: *Journal* ACS (1951) **73**, p. 656.

[16] F. D. Richardson, J. H. E. Jeffes, and G. Withers: *Journal* Iron and Steel Inst. (November 1950) p. 213.

[17] S. S. Todd: *Journal* ACS (1951) **73**, p. 3277.

[18] F. D. Richardson: The Physical Chemistry of Melts. *Inst. of Mining and Metallurgy.* London (1953) p. 83.

Technical Note

Influence of Boron on Hardenability of Steel

by J. C. Fisher

J. C. FISHER, Member AIME, is associated with the Metallurgy Research Dept., Research Laboratory, General Electric Co., Schenectady.

TN 229E. Manuscript, May 14, 1954.

THE following facts are reasonably well established concerning the influence of boron on the hardenability of steel:[1-4]

1—Boron does not appreciably influence the ther-

modynamic free-energy changes occurring during the transformation of austenite.

2—Boron does not influence the temperature range or rate of formation of martensite, lower bainite, carbide, or pearlite.

3—Boron influences only the rate of formation of ferrite or upper bainite that is nucleated at grain boundaries, and that by slowing the nucleation rate or decreasing the number of nuclei, not by altering the growth rate.

4—Increasing the austenitizing temperature from which a steel is quenched can decrease the hardening influence of boron.

5—Increasing the carbon content of a steel decreases the influence of boron.

Since the primary influence of boron is to retard the nucleation of ferrite at grain boundaries, it is possible that this result comes about because of changes in the austenite grain-boundary free energy and the austenite/ferrite interfacial free energy, due to the presence of boron. This possibility will be explored.

Nucleation occurs much more readily at two, three, or four-grain junctions than in the interiors of grains. Grain junctions are favored sites for nucleation because a portion of the austenite grain boundary is eliminated in forming the nucleus, thereby decreasing the increment of interfacial energy associated with nucleus formation. As a matter of fact, if the austenite grain-boundary energy is high enough, and the austenite/ferrite interfacial energy small enough, there will be no increment of interfacial energy associated with nucleus formation at all. In this event, no nucleation barrier exists and ferrite will begin to grow at grain junctions as soon as it is thermodynamically stable.

At a two-grain junction the critical value of the austenite/ferrite interfacial energy is

$$\sigma_{\alpha\gamma} = 0.5\,\sigma_{\gamma\gamma}, \qquad [1]$$

If it is half the austenite grain-boundary energy, two interfaces can be produced for just the energy available in the consumed γ/γ interface, and a lenticular/ferrite platelet can form with no nucleation barrier.

At three and four-grain junctions, the critical values of the austenite/ferrite interfacial energies are[8]

$$\sigma_{\alpha\gamma} = 0.577\,\sigma_{\gamma\gamma} \qquad [2]$$

$$\sigma_{\alpha\gamma} = 0.612\,\sigma_{\gamma\gamma}, \qquad [3]$$

These figures indicate that four-grain junctions are most favored as nucleation sites, three less so, two still less, and grain interiors least favored.

Van Vlack[6] has measured the most probable γ/γ and α/γ interfacial energies, finding

$$\sigma_{\gamma\gamma} = 850 \text{ ergs per sq cm}$$
$$\sigma_{\alpha\gamma} = 630 \text{ ergs per sq cm}$$

from which

$$\sigma_{\alpha\gamma} = 0.74\,\sigma_{\gamma\gamma}, \qquad [4]$$

At first glance this equation indicates that the austenite/ferrite interface has so high an energy that a nucleation barrier will exist even at four-grain junctions. However, remembering that Van Vlack's values of $\sigma_{\gamma\gamma}$ and $\sigma_{\alpha\gamma}$ are most probable values and allowing for some variation in the value of the austenite grain-boundary energy, the magnitude of the variation in $\sigma_{\gamma\gamma}$ required to remove the nucleation barrier can be calculated. (The α/γ interfacial energy will be assumed not to vary. It will have less opportunity to do so, being composed of interfaces between a single ferrite particle, and two, three, or four different austenite grains, tending to average out its value.) At the various types of grain junctions, the following values of austenite grain-boundary energy are required: 1.48, 1.28, and 1.21 times the most probable value for two, three, and four-grain junctions, respectively.

Variations as great as this are not impossibly large. Whether they actually are achieved or not is of secondary importance to the argument. In any event, it is clear that grain junctions are strongly favored sites for the formation of ferrite, and that slight changes in the austenite grain-boundary energy or the austenite/ferrite interfacial energy can have a strong influence on the rate of nucleation or number of nuclei formed. In particular, if boron decreases the austenite grain-boundary energy more than it decreases the austenite/ferrite interfacial energy, nucleation of ferrite can be retarded. If there are grain junctions without nucleation barriers in boron-free austenite, barriers can appear as boron is added. If there are nucleation barriers at each of these sites, they can be raised by the addition of boron.

No bulk free-energy changes are associated with the influence of boron according to this model. Only the rate of nucleation of ferrite is affected. The influence of a higher austenitizing temperature in decreasing the effectiveness of boron reflects the decreased adsorption of boron at high temperatures and is purely an effect of temporarily decreased boron concentration in the region of interest.

The failure of boron to influence the nucleation of carbide or pearlite would indicate, on this model, either 1—that boron decreases the carbide/austenite interfacial free energy about as much as it decreases the austenite grain-boundary energy, or 2—that carbon has already reduced the austenite grain-boundary energy so far that boron has a negligible additional effect.

Acknowledgment

These reflections on the possible role of boron in increasing the hardenability of steel were stimulated by a number of conversations with M. E. Nicholson at the University of Chicago.

References

[1] T. G. Digges and F. M. Reinhart: Influence of Boron on Some Properties of Experimental and Commercial Steels. *Research Paper 1815, Journal of Research* National Bureau of Standards (July-December 1947) **39**.

[2] C. R. Simcoe, A. R. Elsea, and G. K. Manning: An Investigation of the Mechanism by which Boron Increases the Hardenability of Steel. *Report* Battelle Memorial Institute (Aug. 1, 1953).

[3] Boron Steels. Panel on Substitution of Alloying Elements in Engineering Steels, Metallurgical Advisory Board. *Metal Progress* (1951) **60**, p. 81.

[4] Y. Imai and H. Imai: An Investigation on Boron-Treated Steels. I. On the Hardenability of Boron-Treated Medium-Carbon Steels, Especially the Effect of Nitrogen-Content in Steels. *Science Reports* Research Institute Tohoko University (1950) **A2**, p. 260.

[5] P. J. Clem and J. C. Fisher: The Influence of Grain Boundaries on the Nucleation of Secondary Phases. To be published in *Acta Metallurgica*.

[6] L. H. Van Vleck: *Trans.* AIME (1951) **191**, p. 251; JOURNAL OF METALS (March 1951).

Phase Transformations in Titanium-Rich Alloys Of Iron and Titanium

by D. H. Polonis and J. Gordon Parr

High purity alloys of titanium and iron, made by a technique of levitation melting, have been investigated with particular reference to martensite formation and decomposition in the hypoeutectoid range. A preliminary study has been made of the occurrence of the phase corresponding to the structure Ti_2Fe.

MANY of the previous investigations of Fe-Ti alloys have been made using Kroll sponge as a source of titanium; and the subsequent heat treatments to which alloys have been subjected has, on occasion, led to further contamination. Worner,[1,2] who suggested a part of the equilibrium diagram for the binary system, and Wallbaum and coworkers,[3-5] who worked largely with iron-rich alloys, all used Kroll sponge. Van Thyne, Kessler, and Hansen,[6] in their determination of the equilibrium diagram, used iodide titanium for alloys up to 30 pct Fe. Duwez,[7] who measured the M_s temperature of titanium binary alloys with many solute elements, used iodide titanium in all cases except in his work on Ti-Fe alloys for which he used Kroll sponge. The investigation of the supposed phase Ti_2Fe by Rostoker[8] involved the use of iodide titanium. A section of the constitutional diagram, after Van Thyne et al., is reproduced in Fig. 1.

D. H. POLONIS and J. G. PARR, Junior Members AIME, are Graduate Student and Research Associate, respectively, Dept. of Mining and Metallurgy, University of British Columbia, Vancouver, B. C., Canada.

Discussion on this paper, TP 3838E, may be sent, 2 copies, to AIME by Jan. 1, 1955. Manuscript, Apr. 23, 1954. Pacific Northwest Metals and Minerals Conference, May 1954. Chicago Meeting, November 1954.

Frequently too much stress is placed on the effect of contaminants in equilibrium and nonequilibrium systems, but there is no doubt that titanium alloys are extremely susceptible to interstitial atoms. Consequently, the main purpose of the work to be described was to determine those features of phase transformations in pure Ti-Fe alloys that were considered of importance in commercial heat treatments. For, although commercial alloys may not be of high purity, the investigation of a system uncomplicated by traces of impurity is a desirable starting point. Another incidental feature, the occurrence of Ti_2Fe, was also investigated.

The entire study was carried out using powders obtained from alloy ingots, for diffusion reactions are often more rapid in small particles than in bulk specimens. The frequently adopted practice of ascertaining phase constitutions by X-ray analysis of powders, and relating these to the microstructures and mechanical properties of bulk specimens, is considered by the authors to be open to criticism. In the martensite reactions in the Fe-Mn system, for example, it has been shown[9] that similar phase distributions in solid and powder may result in different hardness values and appearance under the microscope; and it is possible that similar anomalies might occur in the Ti-Fe system. Consequently, in order to

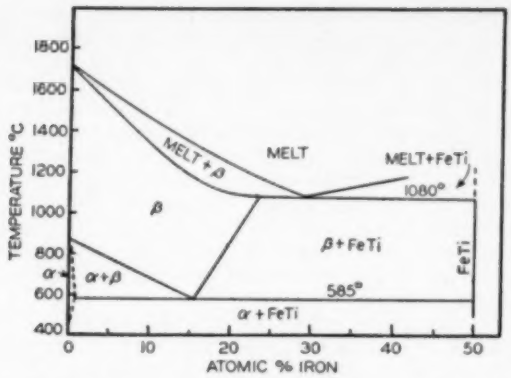


Fig. 1—Ti-Fe phase diagram after Van Thyne, Kessler, and Hansen.[9]

standardize the particle size, all experiments were made with filings or crushed powders of less than 200 mesh. In cases where filings included an oversize fraction, this was discarded; but long-term heat treatments on both size fractions produced equivalent phase distributions and hence indicated that the constitutions of small and large filings were originally identical. This fact was also borne out by microscopic examination. Throughout the experiments every precaution was taken to maintain a high degree of purity in the samples.

Experimental Techniques

Alloy Preparation: Eleven alloys were made in the hypoeutectoid range, from 0.2 to 15.0 atomic pct Fe, as well as three hypereutectoid alloys containing 19.6, 25.3, and 49.6 pct Fe.

The materials used were iodide titanium and Johnson Matthey's electrolytically prepared vacuum-melted iron. A plug of iron was inserted in a hole drilled in a piece of titanium and the metal was melted in levitation by a high frequency coil, the whole being surrounded by a purified argon atmosphere. The technique has been described elsewhere,[10] but it may be mentioned here that each 5 gram mass of metal became molten in about 30 sec and was mixed thoroughly by the stirring effect of the induced currents. The molten material was solidified extremely rapidly by dropping it into a cold copper mold directly beneath the high frequency coil (Fig. 2). There was no trace of reaction or sintering between the mold and alloy, and weighings made before and after melting showed differences of about 1 in 5000, which was the sensitivity limit of the balance used. The ingots were shown to be homogeneous both by microscopic examination and by the indentical X-ray diffraction patterns which were obtained from similarly treated samples of different parts of the same ingot.

As a check on contamination, a piece of pure iodide titanium was melted and cast. Its hardness varied from RF 80 at the center to 90 at the outside. The ingot was melted a second and third time, and there was no change in these hardness values, and, according to accepted standards, therefore, no contamination during melting. Although the usual hardness value quoted for pure as-cast titanium is 70 to 75 RF, the difference is believed to be due to the greater rate of solidification and cooling in the levitation-melted material. Internal strains and a smaller grain size may well account for the discrepancy.

Heat Treatments—*Quenching:* Although the actual rate has not been determined, the critical quenching rate from the β phase (body-centered cubic) to produce martensitic α' (close-packed hexagonal) is not very great. The hazards of contamination ruled out the possibility of water-quenching unenclosed powders. And as it was sometimes observed that titanium appeared to reduce silica at temperatures above 800°C (an observation which agrees with the available free-energy data), it was felt inadvisable to quench sealed-off specimens. Consequently, a gas-quenching furnace was designed in which the specimen was held in an atmosphere of argon (twice purified over hot calcium and copper) and blown out by a stream of cold gas. The powder was held in a small molybdenum boat, with which no reaction occurred, heated to temperature in 7 min, soaked, and blown out. The powder sintered lightly during this treatment, and the cooling rate therefore was comparatively slow. It was assessed at about 200°C per sec. However, a second arrangement was devised in which the specimen was rotated during its heat treatment; therefore it had no opportunity to sinter. The cooling rate on quenching was consequently much more rapid, since individual particles were cooled by the gas stream. However, the amount of martensitic α' produced in a given alloy was the same in both techniques and, because of its comparative simplicity, the first method was adopted.

At the time of these experiments, helium was not readily available. Although it is realized that helium would allow a more rapid quench, it was quite clear that the rate attained by the argon quench was greater than critical.

Tempering: The alloys were tempered at one temperature only, 570°C, for 10, 100, and 1000 hr. Even at this temperature, which is fairly close to the eutectoid horizontal of 585°C (Van Thyne et al.), decomposition rates of retained β were slow. However, it is hoped that further experiments will be conducted at other temperatures in order to gather information to allow an analysis of the decomposition kinetics of the β phase.

For tempering treatments, the quenched powders were sealed in vacuo in silica tubes. The same specimens were used for successive heat treatments,

Fig. 2—Levitation-melting unit.

Fig. 3—High temperature X-ray diffraction unit.

since no phase transformations occur on cooling from the tempering temperature.

Assessment of the Constitution of Heat-Treated Powders: Plots made by Geiger-counter spectrometer of the intensities of diffraction lines from the phases α (close-packed hexagonal), β (body-centered cubic), and FeTi (body-centered cubic) allowed an assessment to be made of their quantities. The method used is similar to that already described in connection with Fe-Mn alloys.[11, 12] Two difficulties arise: 1—In order that the method be reliable, it is desirable to use neighboring diffraction lines. Strong lines from each of the phases, 101α, 110β, and 110 FeTi, occur within the range of θ values 19°to 22°, using Cu Kα; but the first two of these lines tend to overlap. However, by using the 002α line, which occurs at approximately 19°, an assessment of the amount of α could be made and the strength of the 101α line computed. The intensity of the 110β line then could be assessed by difference. 2—At low angles, the parameter difference between martensitic α' and equilibrium α in tempered structures is not detectable. Consequently, the values obtained for the amount of hexagonal phase include both structures. Therefore, while it is possible to state the amount of α' produced on quenching (for all the hexagonal phase is martensitic) the amount of equilibrium α formed on tempering cannot be calculated because the intensities of diffraction lines from the hexagonal phases increase due to the precipitation of equilibrium α, but decrease due to the decomposition of α'. In alloys in which no α' is formed on quenching, all the hexagonal phase subsequently produced during tempering is equilibrium α. This should be borne in mind when reading the α curves in the graphs illustrating the effect of tempering quenched alloys.

Determination of M_s Temperatures: The methods available for M_s determination fall into two groups, cooling-curve techniques and metallographic techniques. Although it is hoped that the first method will be used eventually, the measurements made up to date have been by metallographic methods.

If a low iron alloy is cooled at a greater than critical rate through the M_s temperature, martensitic α' is formed. If the quench is interrupted just above M_s, equilibrium α would be expected to precipitate from the untransformed β phase. The two forms of α are readily distinguishable under the microscope.

The alloy powder was soaked at 1000°C and pulled into a second zone in the furnace tube where it could be maintained at a lower temperature (subsequently referred to as the holding temperature). This heat treatment was carried out in an atmosphere of purified argon. The powder was then quenched in air, since the holding temperature did not exceed 350°C and oxidation on cooling from this temperature is imperceptible and restricted to a layer of only a few atoms.

It was appreciated that the cooling rate from soaking to holding temperature might be sufficiently slow to allow the precipitation of equilibrium α on cooling through the α-β phase boundary. But this possibility was checked by maintaining the alloy at the holding temperature for different time intervals before quenching to room temperature. If equilibrium α had formed on cooling to the holding temperature, then its extent would not be affected by holding time; but, if it were produced isothermally at the holding temperature, its amount would increase at longer holding times. That the second of these two possibilities occurred is shown in illustrations that are referred to later. The possibility that equilibrium α is formed by the breakdown of martensite already produced is also ruled out, for the typical martensitic structure once formed is extremely persistent up to the eutectoid temperature. Finally, the fact that the nonmartensitic phase was precipitated equilibrium α and not stabilized β (which might occur at sub-M_s temperatures) was ascertained by X-ray diffraction photographs.

High Temperature X-ray Diffraction Studies: In order to make a few spot checks at certain parts of the equilibrium diagram and to determine the eutectoid temperature, a small furnace was constructed to surround a powder specimen held at the centre of the X-ray goniometer. The detailed construction of the furnace is to be described in another paper; a photograph is shown in Fig. 3. The furnace atmosphere was of helium, purified by passage through activated silica gel at liquid-oxygen temperature. This atmosphere was maintained, with a slow stream of helium continually passing through

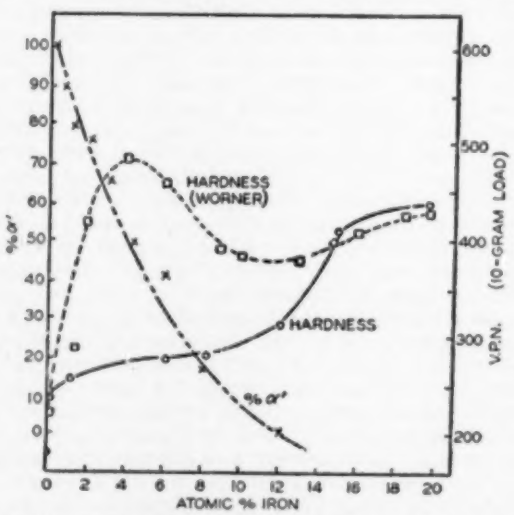


Fig. 4—Constitution and hardness of alloys quenched from 1000°C.

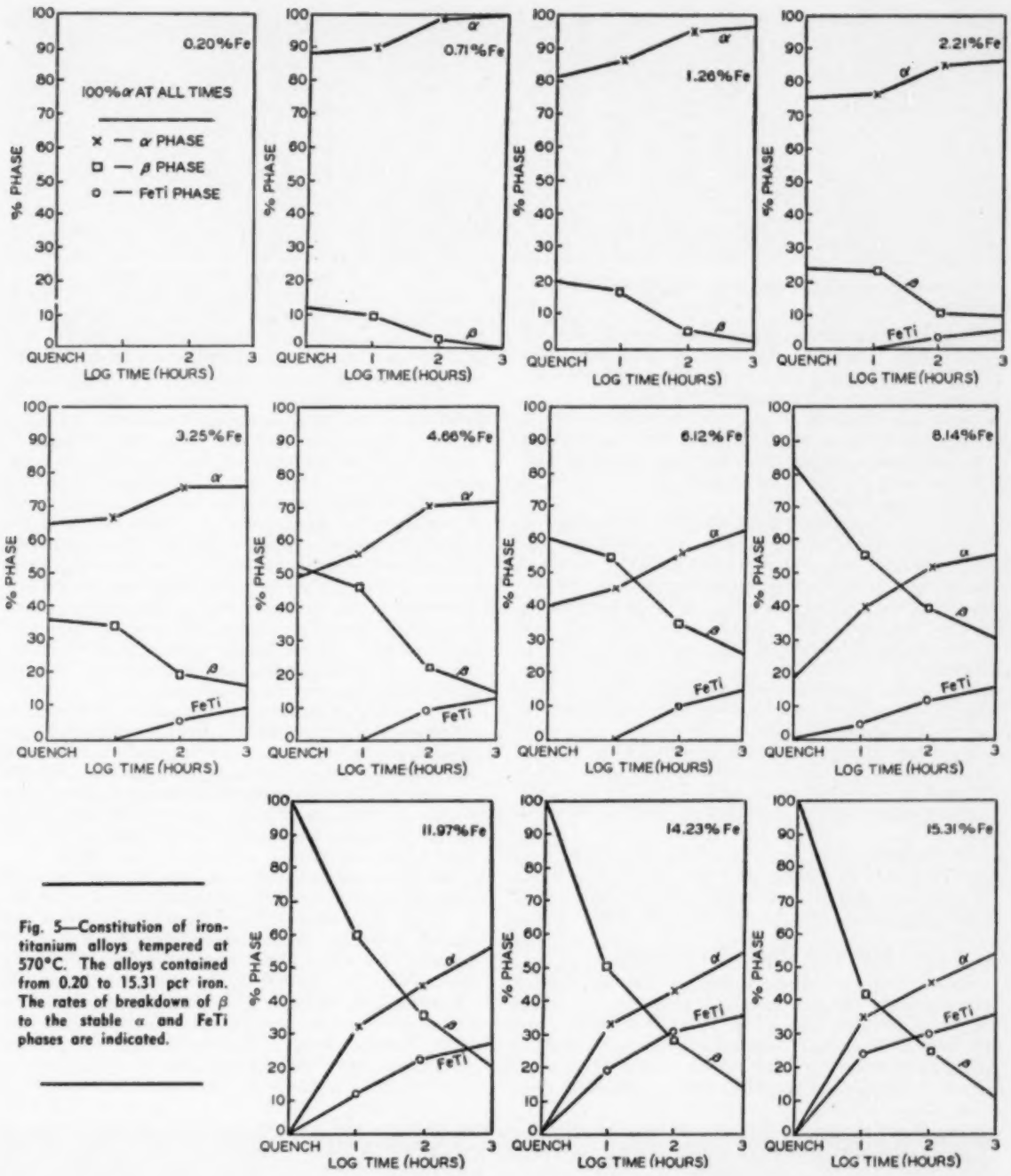


Fig. 5—Constitution of iron-titanium alloys tempered at 570°C. The alloys contained from 0.20 to 15.31 pct iron. The rates of breakdown of β to the stable α and FeTi phases are indicated.

the furnace for several hours before the specimen was heated. Occasionally, the silica-gel purification tube was preceded by a hot calcium and copper train; but this was eventually considered to be unnecessary.

Hardness Measurements: Hardness measurements were made on heat-treated powders, mounted in plastic, with a Bergsman microhardness tester using loads of 10 and 25 grams applied to a diamond indenter. In each sample the hardness of ten or more particles was measured, and the variation was not more than 10 pct.

Contamination during Heat Treatment: Although the use of hardness values in assessing contamination is open to criticism, the method is generally accepted. Therefore, it is felt that some hardness checks made on pure titanium subjected to certain heat treatments are of interest. Although the hardness values given are related to oxygen contamination, the figures really represent a combined oxygen and nitrogen content.

1—Iodide-titanium bar stock gave RF 72 (60 kg load). This is approximately equivalent to 75 Vhn.

2—Microhardness test (10 and 25 gram loads) for iodide-titanium bar stock gave 110 Vhn. The fact that the hardness value is greater when a small load is used probably arises because the very small penetration is affected by surface irregularities. As

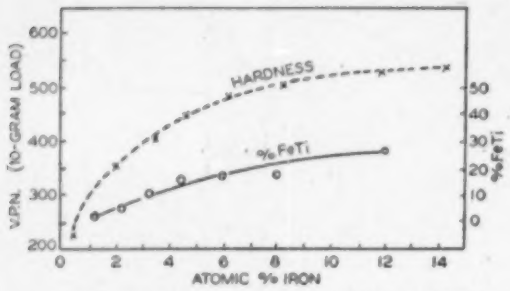


Fig. 6—Hardness of alloys tempered at 570°C for 1000 hr.

all other hardness tests were made with 10 and 25 gram loads, it seems reasonable to take the value of 110 Vhn as the hardness of uncontaminated titanium under the conditions of the test.

3—Iodide-titanium filings were heated to 1000°C in purified argon in the quenching furnace, held for 1 hr, and then slowly cooled. The Vhn was 120. The increase of 10 Vhn reflects an increase of about 0.03 atomic pct O.

4—Iodide-titanium filings were heated to 1000°C in vacuo in a sealed silica tube, held for 1 hr, and then slowly cooled. Hardness values were scattered and gave the following approximate distribution: 45 pct, 120 Vhn with 0.03 pct O pickup; 50 pct, 120 to 180 Vhn with 0.03 to 0.25 pct O pickup; and 5 pct, 180 to 250 Vhn with 0.25 to 0.7 pct O pickup. Contamination is believed to be due to the reduction of silica by titanium at points of contact. This view is supported by the grey appearance of the inside of the quartz tube.

5—Iodide-titanium filings were heated to 1000°C in the high temperature X-ray furnace, held for 1½ hr, and then cooled in 15 min. The Vhn was 150. The increase of 40 Vhn reflects an increase of about 0.20 pct O.

Scratch hardness tests made across individual particles of heat-treated titanium produced a mark of constant width, implying that the hardness and hence the composition of each particle was uniform.

Results for Hypoeutectoid Alloys

Quenched Structures: As will be seen from Fig. 4, some α' is produced in quenched powders containing up to 12 pct Fe. This contrasts with Worner's observations that water-quenched alloys containing more than 3.8 pct Fe are retained as β solid solutions. The fact that Worner's alloys contained other elements, and were quenched from 950°C, might account for the difference.

A further discrepancy between these results and Worner's is the absence of the pronounced peak in the hardness of quenched specimens which Worner finds at 5 pct Fe (see Fig. 4). This difference may be due to the oxygen and nitrogen content of Worner's alloys, or to the fact that, in his experiments, hardness measurements were made on bulk samples, while in this work powders were used. That the size of the specimen undergoing a martensitic reaction may affect hardness values considerably has already been demonstrated.[9]

Under the microscope, martensitically produced α' in the powder specimens shows the typical Widmanstaetten pattern of strain lines. (Fig. 7.) On the whole, structures are similar to those already published in the papers referred to earlier.

Tempered Structures: The effect of tempering at 570°C is summarized in the graphs shown in Fig. 5, which indicate rates of breakdown of β to the stable α and FeTi phases. It will be observed that the curves of β breakdown increase in slope as the iron content increases, presumably because there is more β available for decomposition. Further, while the logarithmic plots of amount of β for alloys containing more than 6 pct Fe approximate to smooth curves, alloys containing less than 6 pct Fe show a discontinuity at the point on the time axis at which FeTi begins to appear. This may be associated with an incubation period for the formation of FeTi, for after its appearance the slope of the curve, amount of β vs log time, shows an increase.

After 1000 hr tempering, the ratios of FeTi:α in all alloys above 1 pct Fe are greater by about 50 pct than is to be expected from calculations made by applying the lever rule to the equilibrium diagram. Presumably the presence of retained β throws the system out of equilibrium, and further tempering would favor the formation of α, in order to produce equilibrium mixtures.

Hardness measurements were made on the specimens tempered for 1000 hr. These are reproduced in Fig. 6. The steady rise in hardness may be associated with the increased amounts of FeTi produced in the alloys. It will be noticed that the hardness of the 0.2 pct Fe alloy remains substantially unchanged on tempering; and its appearance under the microscope remains martensitic.

M. Temperatures: The metallographic method employed may be applied only to alloys whose martensitic structure is readily distinguishable

Fig. 7—1.26 pct Fe alloy quenched from 1000°C. Entirely martensitic. Etched in aqueous 1 pct HF, 1 pct H_2O_2. X800. Area reduced approximately 50 pct for reproduction.

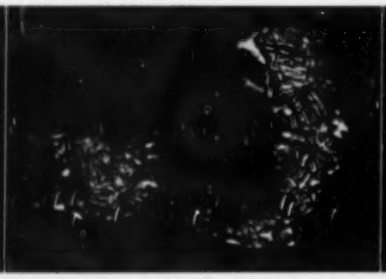


Fig. 8—0.2 pct Fe alloy quenched from 1000°C, held at 300°C for 1 min, then cooled to room temperature. Martensitic α' with a trace of light-etching equilibrium α. Etched in aqueous 1 pct HF, 1 pct H_2O_2. X800. Area reduced approximately 50 pct for reproduction.

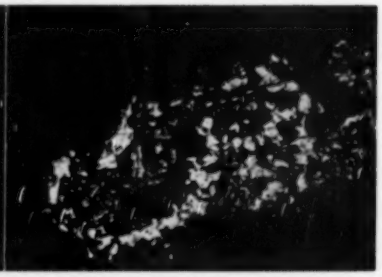


Fig. 9—0.2 pct Fe alloy quenched from 1000°C, held for 100 min at 300°C. The amount of equilibrium structure has increased. Etched in aqueous 1 pct HF, 1 pct H_2O_2. X800. Area reduced approximately 50 pct for reproduction.

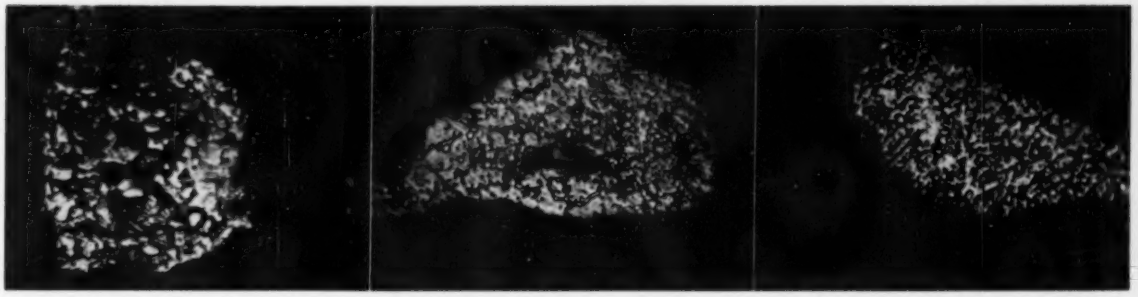


Fig. 10—0.2 pct Fe alloy quenched from 1000°C, held for 500 min at 300°C. There is further increase in the amount of equilibrium structure. Etched in aqueous 1 pct HF, 1 pct H_2O_2. X800. Area reduced approximately 50 pct for reproduction.

Fig. 11—25 pct Fe alloy as-cast. FeTi in a β matrix. Etched in aqueous 1 pct HF, 1 pct H_2O_2. X800. Area reduced approximately 50 pct for reproduction.

Fig. 12—25 pct Fe alloy held at 1000°C for 1 hr. Largely Ti_2Fe and β. Etched in aqueous 1 pct HF, 1 pct H_2O_2. X800. Area reduced approximately 50 pct for reproduction.

from the equilibrium structure. Where large quantities of β are retained, differentiation is difficult, since the quenched structure does not show a very pronounced strain pattern. Consequently, experiments so far have been limited to alloys containing less than 3 pct Fe.

By the method described in a previous section, the M_s temperature of a 0.20 pct alloy was found to be 280°±15°C. Micrographs reproduced in Figs. 8 to 10 show the effect of isothermal holding at 300°C; and it will be noted that greater quantities of equilibrium α (the light-etching constituent) are precipitated as the holding time increases.

Alloys containing more iron show a higher M_s temperature, rising to about 350°C at 2.2 pct Fe.

These results conflict with the measurements made by Duwez, whose values for M_s fell from 800°C for a 1 pct alloy to 400°C for a 4.5 pct alloy. Apart from the fact that Duwez used Kroll sponge in making his alloys, no other explanation can be offered for this marked difference, unless M_s is sensitive to the cooling rate. A slow cooling rate (such as was used in the experiments described here) may lead to the successive stabilization of β as the temperature falls, and result in depressed M_s.[13] On the other hand, some martensitic reactions show a higher M_s as the cooling rate decreases.[14] Although Duwez showed that there was little variation in M_s with cooling rates of 100° to 10,000°C per sec, the rate employed in authors' experiments is believed to have averaged about 50°C per sec. However, the rate of cooling through temperatures of 800°C (M_s according to Duwez) would be considerably faster than this average, and would fall between Duwez's limits of 100° to 10,000°C per sec. Consequently, if M_s is about 800°C for a 0.2 pct alloy, there is no doubt that martensite should have been produced in alloys cooled to 300°C; and holding at this temperature would have little subsequent effect. The micrographs, however, demonstrate the continued precipitation at 300°C of a phase which X-ray analysis shows to be α; hence 300°C is above the M_s temperature. A series of experiments bracketed the M_s at 280°C in this alloy. Further experiments are being carried out on this aspect of the transformation.

Eutectoid Temperature: It is valuable to know the eutectoid temperature with some accuracy, since it represents the maximum temperature at which quenched structures may be tempered safely. It was determined by high temperature X-ray measurements on an alloy containing 12.0 pct Fe. Initially the filings were composed largely of retained β with some α'. On heating to temperatures about 500°C, reflections from FeTi appeared. The continued appearance of this phase was observed by intensity measurements of its 110 reflection. Up to a temperature of 600°C, the amount of FeTi increased. Repeated scans were made over a range of θ in which $FeTi_{110}$, β_{110}, and α_{101} appeared. At 625°C an equilibrium condition seemed to have been established in which there was no change in the quantities of the phases present over a period of three hours. At higher temperatures the FeTi line began to disappear.

A second determination was made on cooling from the β range. The specimen in a wholly β condition at 1000°C was cooled to 600°C. A small θ range in which the $FeTi_{110}$ line occurred was scanned repeatedly. After 20 min at 600°C, a broad low-intensity reflection was observed, which, during the subsequent two hours, resolved into a fairly sharp peak.

Diffusion reactions in the system Ti-Fe are slow, and consequently this temperature, 600°C, at which FeTi appeared, must have been below the eutectoid temperature. Using this result in conjunction with the previous one, the eutectoid temperature is assessed at 625°±10°C. This value is 25° to 40°C higher than the customarily accepted one.

Results for Hypereutectoid Alloys

The three alloys, 19.6, 25.3, and 49.6 pct Fe have been examined by X-ray diffraction and microscopic techniques after heat treatment at 1000°C. The 25 pct alloy showed the phase that has been designated $FeTi_2$ and, after holding for 1 hr at 1000°C, consisted almost entirely of $FeTi_2$ and β. The 20 pct alloy showed predominantly β with some $FeTi_2$, while the 50 pct alloy consisted of FeTi with a little $FeTi_2$. As the crystal structure of $FeTi_2$ is not known with certainty, no quantitative estimates can be made of its amount.

FeTi: To supplement Worner's account of this structure, accurate measurements were made of its lattice parameter and density. A parameter of 2.978Å±0.001 compares with Worner's value of 2.97Å. Assuming the structure to be body-centered cubic, as diffraction photographs indicate, and taking a value of 6.025×10^{23} for Avogadro's number, the density should be 6.52 grams per cu cm. The density measured by a water-displacement method is 6.50 grams per cu cm ±0.03. Slowly cooled powders were examined carefully for the appearance of

superlattice lines. X-ray photographs were taken using Cr $K\alpha$ to depress the scattering factor of iron and hence increase the differential between the atomic scattering of titanium and iron. No ordering was observed.

Ti_4Fe: Although this phase has been reported by Laves and Wallbaum[4] and by Duwez and Taylor,[10] no other investigators have confirmed its existence. Rostoker[9] made up alloys corresponding to the compositions Ti_4Fe, Ti_4Fe_2O, and Ti_4Fe_2O and detected a phase corresponding to Ti_4Fe only in the alloys containing oxygen. In further work[10] Rostoker established the isothermal section at 1000°C of the system Ti-Fe-O. The phase corresponding to Ti_4Fe occurs with β and FeTi in a large area whose boundary nearest to the Ti-Fe coordinate is at 2 pct O. Hence it would appear that at 1000°C the phase corresponding to Ti_4Fe is produced only when there is 2 pct or more oxygen.

However, in this work alloys contained considerably less oxygen than this minimum, and some experiments were conducted on the 25 pct alloy to ascertain the reason for the occurrence of the phase. Its structure is complex, approximating to a face-centered cube with cell side 3.78Å. But the true structure cell is made of 27 such units, so that the cell is a cube side of approximately 11.34Å.

Although the phase was completely absent from the as-cast specimen of the 25 pct alloy (which contained only β and FeTi), it formed on heating at 1000°C. The phase was produced in powders that were heat treated in purified argon, purified helium, and sealed in vacuo. High temperature X-ray examination showed that the phase was present at 1000°C, for at this temperature diffraction lines of FeTi, Ti_4Fe, and β were observed.

In order to account for the difference between these results and Rostoker's, the following explanation is tentatively offered: The as-cast 25 pct Fe alloy made by levitation melting shows a very finely dispersed precipitate of FeTi in a β matrix. (Fig. 11). These components react in crushed-powder specimens to form Ti_4Fe and β on suitable heat treatment. (Fig. 12.) In cast alloys that are more slowly cooled, as larger samples made by arc-melting could be, the FeTi would tend to segregate and the reaction to form Ti_4Fe would be much slower. Even so, it is believed that prolonged heating at 1000°C would produce some Ti_4Fe in alloys of appropriate composition; but no experiments appear to have been conducted on these lines. The effect of oxygen is to increase the range of stability of the Ti_4Fe, and probably to accelerate the reaction producing it.

The three hypereutectoid alloys were tempered at 570°C. The small quantity of Ti_4Fe in the 50 pct alloy had disappeared at 100 hr treatment, leaving a structure of entirely FeTi. The 20 pct alloy showed predominantly β after 100 hr tempering, while the 25 pct alloy showed FeTi and β with a little remaining Ti_4Fe after 1000 hr. It would appear that the phase Ti_4Fe is stable only above the eutectoid temperature; but its range of stability and the consequent modification of the Ti-Fe equilibrium diagram have not yet been investigated.

Summary

High purity alloys of titanium with iron, made by a technique of levitation melting, have been investigated. Alloys containing up to 12 pct Fe may produce some martensitic α' on quenching in argon from 1000°C, but this is not accompanied by a marked increase in hardness in powder specimens. Retained β is slow to decompose at a temperature 50°C below the eutectoid, but rates are greater at higher compositions. The appearance of FeTi on tempering is usually accompanied by an increase in the slope of the curve of β/log time. Tempered alloys have hardness values that rise as the FeTi content increases.

The M_s temperature of a 0.20 pct Fe alloy has been determined by metallographic methods to be 280°±15°C; the M_s temperature rises to 350°C for a 2.2 pct Fe alloy.

High temperature X-ray examination has established the eutectoid temperature at 625°±10°C.

The density and parameter of FeTi have been found to be 6.50 grams per cu cm ±0.03 and 2.978Å±0.001. The structure shows no ordering.

Although previous work has implied that the structure corresponding to Ti_4Fe occurs only in alloys containing oxygen, this fact is refuted by results obtained in this work. The structure Ti_4Fe may be produced by the reaction of FeTi and β in crushed powder from the as-cast alloy when it is appropriately heat treated, and has been observed by high temperature X-ray techniques at 1000°C. The phase decomposes below the eutectoid temperature.

Acknowledgments

The authors wish to acknowledge the contribution of the Defence Research Board of Canada in furnishing the funds which enabled this study of titanium alloys to be carried out (Project No. 425). A fellowship awarded by the International Nickel Co. of Canada to one of the authors is gratefully acknowledged.

We are grateful to Professor F. A. Forward for his encouragement, and to Professor W. M. Armstrong, Mr. H. G. V. Evans, and our other colleagues for their stimulating discussions. We are particularly indebted to Mr. R. G. Butters who has been responsible for the design and construction of much of the apparatus.

References

[1] H. W. Worner: *Journal* Institute of Metals (1951) **79**, No. 3, p. 173.
[2] H. W. Horner: *Journal* Institute of Metals (1951) **80**, No. 5, p. 213.
[3] H. Witte and H. J. Wallbaum: *Ztsch. Metallkunde* (1938) **30**, p. 100.
[4] H. J. Wallbaum: *Archiv. Eisenhuttenwesen* (1938) **12**, p. 33.
[5] F. Laves and H. J. Wallbaum: *Naturwissenschaften* (1939) **27**, p. 674.
[6] R. J. Van Thyne, H. D. Kessler, and M. Hansen: *Trans.* ASM (1952) **44**, p. 974.
[7] P. Duwez: *Trans.* ASM (1953) **45**, p. 934.
[8] W. Rostoker: *Trans.* AIME (1952) **194**, p. 209; JOURNAL OF METALS (February 1952).
[9] J. G. Parr: *Journal* Institute of Metals (1952) **81**, p. 214.
[10] D. H. Polonis, R. G. Butters, and J. G. Parr: *Research* (1954) **7**, p. 10s.
[11] J. G. Parr: *Journal* Iron and Steel Institute (1952) **171**, p. 137.
[12] J. G. Parr: *Research* (1953) **6**, p. 37s.
[13] E. R. Morgan and T. Ko: *Acta Metallurgica* (1953) **1**, p. 36.
[14] C. L. M. Cottrell: *Journal* Iron and Steel Institute (1954) **176**, p. 269.
[15] P. Duwez and J. Taylor: *Trans.* AIME (1950) **188**, p. 1173; JOURNAL OF METALS (September 1950).
[16] W. Rostoker: Private communication.

Quantitative Substructure and Tensile-Property Investigations of Nickel Alloys

by Betsy Ancker and Earl R. Parker

The small-angle dislocation-boundary density of nickel and some of its alloys was investigated as a function of strength. It was found that the strength is a linear function of the density for pure nickel and for Ni-Ti alloys, but not for Ni-Co alloys. The results are interpreted in terms of dislocation theory.

THE processes by which metals deform are still poorly understood, even though there has been a concentrated research effort in this field during the past fifteen years. Single crystals and individual grains in polycrystalline aggregates often develop a subgrain structure when deformed. Substructure formation occurs as a result of local plastic bending and its development is augmented by annealing at moderate temperatures.[1] Investigations made to date, largely qualitative in nature, have shown that the substructure is composed of a network of small-angle boundaries. The purpose of the present paper is to present some quantitative data on substructure (obtained by X-ray techniques) and to correlate the results with the strength of the metal in which the substructure existed. Nickel and some of its alloys were used for the experiments. The presence of certain kinds of foreign atoms greatly alters the formation and effect of the substructure.

Polycrystalline tensile specimens were prepared from high purity nickel, two Ni-Ti alloys, and two Ni-Co alloys; the composition of these materials is given in Table I. Tensile specimens having a reduced section ¼ in. diam by 3 in. long were machined from materials which were refined, cast, forged, and swaged in the authors' laboratories. The specimens were annealed for ½ hr at 1150°C, a treatment which produced grains having a diameter of approximately 0.5 mm. This rather large grain size was used to facilitate later examination of the substructure. Duplicate specimens of pure nickel and of each alloy were strained various controlled amounts in tension at room temperature and recovered for 1 hr at 800°C. (This strain induced prior to recovery will be referred to hereafter as the "prestrain.") One of each of the recovered specimens was pulled in tension at room temperature to obtain the stress-strain curves. The other bar was cross-sectioned in the gage length, carefully polished, and deeply etched to remove all cold-worked material. This specimen was then examined by the X-ray microscopic technique[2] to determine the nature and magnitude of the substructure. The use of duplicate specimens permitted a direct correlation between tensile properties and substructure characteristics.

B. ANCKER and E. R. PARKER, Member AIME, are Research Physicist and Professor of Metallurgy, respectively, Minerals Research Laboratory, University of California, Berkeley, Calif.

Discussion on this paper, TP 3845E, may be sent, 2 copies, to AIME by Jan. 1, 1955. Manuscript, Apr. 20, 1954. Chicago Meeting, November 1954.

X-ray microscopic studies were made with an apparatus constructed to have an average resolving power of about 4 microns. The monochromatic radiation reflected from grains which were oriented favorably was recorded on fine-grain photographic plates. These were examined at X100 which represents the true magnification, since there was essentially none in the X-ray system. This permitted detection of angular differences between subgrains of as little as 10 min. A very large number (over 100) of grains for each specimen was examined at this magnification and the number of substructure boundaries per grain was tabulated. An accurate relative measure was obtained under fixed experimental conditions, e.g., same grain size, resolving power, magnification, etc. The greatest source of error lay in counting the subboundaries, but this proved to be surprisingly small when repeated counts were made by different investigators. Furthermore, as the subsequent analysis indicates, whatever human error was introduced was not significant.

Analysis of the Substructure Data

When the number of substructure boundaries per grain was plotted against the frequency, skew (asymmetrical) distribution curves of the type shown in Fig. 1 were obtained. These are smoothed curves for the 1 atomic pct Ti in nickel alloy subjected to various prestrains and are representative of the curves obtained with all the metals. Since skew-distribution curves cannot be analyzed by elementary statistical methods, detailed investigations were undertaken in order to 1—test the consistency

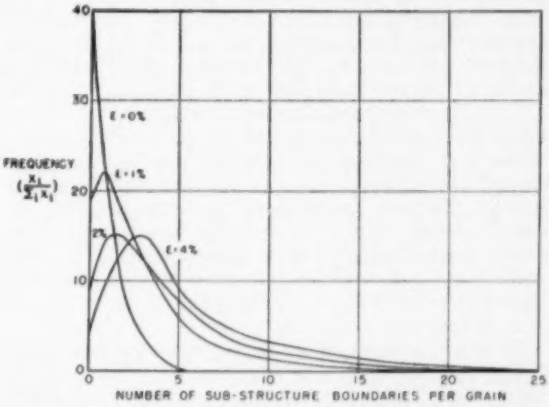


Fig. 1—Skew distribution curves for 1 atomic pct Ti in nickel with varying prestrains.

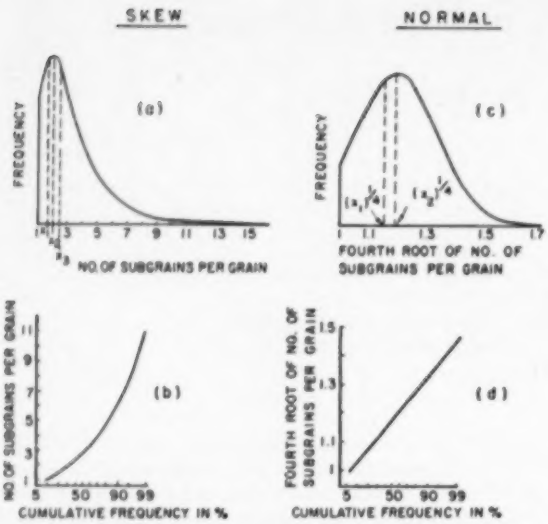


Fig. 2—A representative skew frequency distribution transformed into the corresponding normal distribution.

Table I. Tabulation of the Substructure Parameters

Prestrain, Pct	Alloy	Substructure Parameters		
		Mode	Median	Mean
0	99.95 pct Ni	0.16	0.34	1.18
	1 atomic pct Ti in Ni	0.54	0.94	1.78
	2 atomic pct Ti in Ni	0.92	1.51	2.62
	5 atomic pct Co in Ni	0.70	1.36	2.24
	20 atomic pct Co in Ni	0.79	1.78	2.85
1	99.95 pct Ni	0.59	1.91	3.42
	1 atomic pct Ti in Ni	0.87	2.52	4.01
	2 atomic pct Ti in Ni	1.07	2.99	4.51
	5 atomic pct Co in Ni	1.05	2.56	3.96
	20 atomic pct Co in Ni	1.56	3.42	4.79
2	99.95 pct Ni	1.15	3.03	4.73
	1 atomic pct Ti in Ni	1.59	3.84	5.75
	2 atomic pct Ti in Ni	1.69	4.07	5.90
4	1 atomic pct Ti in Ni	2.45	4.80	6.25
	2 atomic pct Ti in Ni	2.77	5.63	7.70
	5 atomic pct Co in Ni	1.27	3.65	5.44
	20 atomic pct Co in Ni	1.59	4.80	6.93

of the data and 2—obtain parameters suitable for describing the distribution curves and for comparing data obtained from different specimens.

A modification of the Wilcoxon test* was used to verify or reject the hypothesis that two sample distributions came from the same general distribution. This test was used to show not only that various samples from one specimen all came from the same distribution, but also that samples from different alloys or those having different prestrain histories did not come from the same distribution. In every case significant differences in the distribution of the number of substructure boundaries per grain were shown to be present among the various specimens having different amounts of prestrain or different alloy content. Only rarely were there indications that various samples from one specimen did not come from the same distribution. The inconsistent data represented only a small part of the total and was discarded.

In order to analyze and compare data from different specimens, it was necessary to transform the skew-distribution curves into normal (Gaussian) ones. The cumulative-frequency plot (Fig. 2b) corresponding to a representative skew-frequency distribution (Fig. 2a) is a curve. By using a suitable transformation function, such a skew distribution may be made normal as illustrated by Fig. 2c and its cumulative frequency is then represented by a straight line (Fig. 2d). It was found that the skew distributions of the substructure boundaries per grain for various strains and alloys could be transformed into normal distributions by the use of the fourth-root function. Suitable parameters for describing the original distribution can be calculated or obtained easily from the transformed cumulative-frequency plot. These parameters (see Figs. 2a and 2c) are: mode, the number of subgrains per grain which occurs most frequently in the distribution, $x_1 = (\xi^{1/4} - 3\sigma^2)^4$; median, half the grains have this number of subgrains or more, $x_2 = \xi$; and mean, the average number of subgrains per grain, x_3 where σ is standard deviation of transformed distribution.

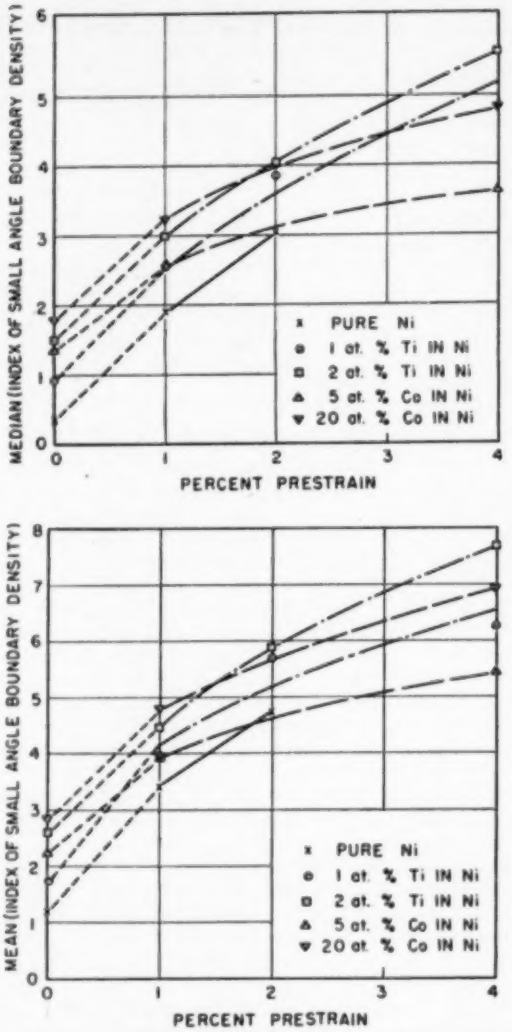

MEDIAN (INDEX OF SMALL ANGLE BOUNDARY DENSITY)
PERCENT PRESTRAIN
x PURE Ni
• 1 at. % Ti IN Ni
□ 2 at. % Ti IN Ni
△ 5 at. % Co IN Ni
▽ 20 at. % Co IN Ni
MEAN (INDEX OF SMALL ANGLE BOUNDARY DENSITY)
PERCENT PRESTRAIN
x PURE Ni
• 1 at. % Ti IN Ni
□ 2 at. % Ti IN Ni
△ 5 at. % Co IN Ni
▽ 20 at. % Co IN Ni

Fig. 3—The relationship between prestrain and density of substructure boundaries as indicated by median (top) and mean (bottom).

Experimental Results

Results obtained from the statistical analysis of the substructure determinations are summarized in Table I, while the cumulative-frequency curves from which these data were derived are contained in

* See Appendix A for details, see p. 1161.

Appendix B. All three parameters (mode, median, and mean) vary in the same general manner with prestrain and alloy content. The median and mean, as indices of small-angle boundary density, are plotted against the amount of prestrain in Fig. 3 for pure nickel and the four alloys. A similar plot of mode, as substructure parameter, vs prestrain is shown in Fig. 4. The substructure formation behaviors of the Ni-Ti and Ni-Co systems are revealed most graphically by using the mode as the index of small-angle boundary density.

Room-temperature tensile properties for pure nickel and for each alloy, with various amounts of prestrain, are shown in Figs. 5 through 9. The flow stress at a given strain increases regularly with increasing prestrain for all of the alloys tested. This is shown more clearly by Fig. 10, in which the flow stress at 0.25 pct strain is plotted against the amount of prestrain. The effects of increasing concentrations of foreign atoms on the substructure density and on the strength of nickel are shown in Fig. 11. The flow stress increased linearly with both titanium and cobalt content; the amount of substructure in the titanium alloys increased in a comparable manner. The substructure density in the cobalt alloys, however, rapidly approached a maximum. This evidence showed that a simple correlation between substructure density and strength is not generally valid.

Discussion

Several mechanisms of work hardening operate to increase the strength of a metal subjected to plastic deformation at low temperatures. Before the effects of the individual factors can be isolated, measured quantitatively, and understood, the metals to be used in the investigation must be put into comparable initial states. The same strain history associated with the preparation of the specimens, plus the same original annealing treatment (1150°C for ½ hr), yielded a different density of substructure for each alloy. This (variable) small-angle boundary density and the corresponding strengths of the alloys as compared with that of the base metal may be attributed to the presence of the foreign atoms (see Fig. 11). However, the nature of the subboundaries after an 1150°C anneal is not the same as after an 800°C treatment (see Fig. 12). Consequently, all specimens were strained 1 pct and heated to 800°C to produce a base state in which the subboundary structure was similar for each specimen. The number of subboundaries was, of course, different for each metal but this was of minor importance in the room-temperature deformation studies because comparison between substructure and strength was made on the basis of differences produced by additional increments of strain beyond the initial 1 pct.

Substructure boundaries are introduced by local plastic bending. Regions of a crystal which have

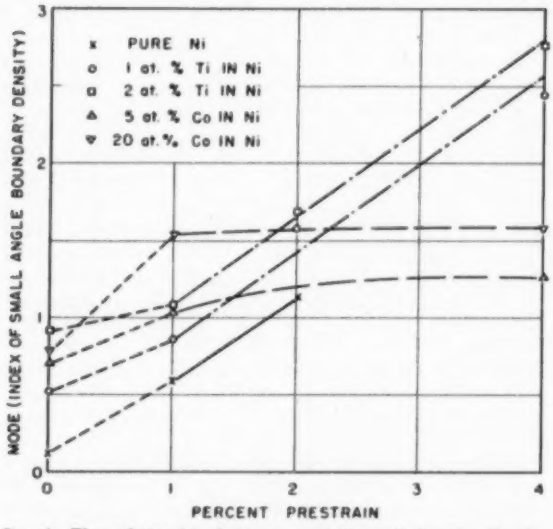


Fig. 4—The relationship between prestrain and density of substructure boundaries as indicated by the mode.

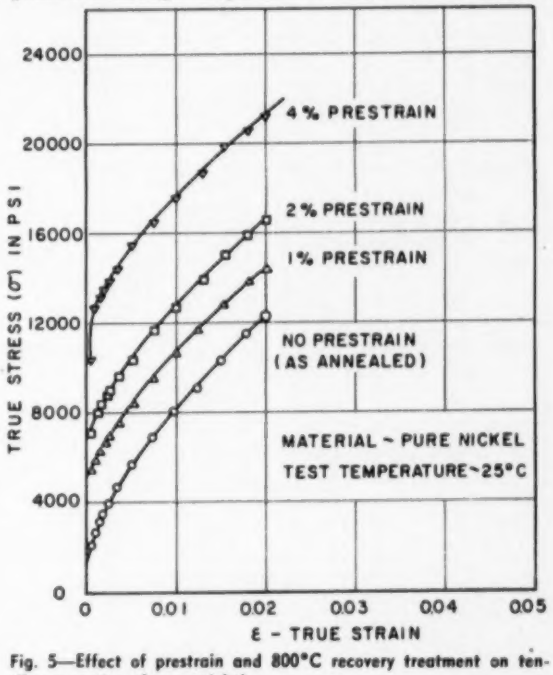


Fig. 5—Effect of prestrain and 800°C recovery treatment on tensile properties of pure nickel.

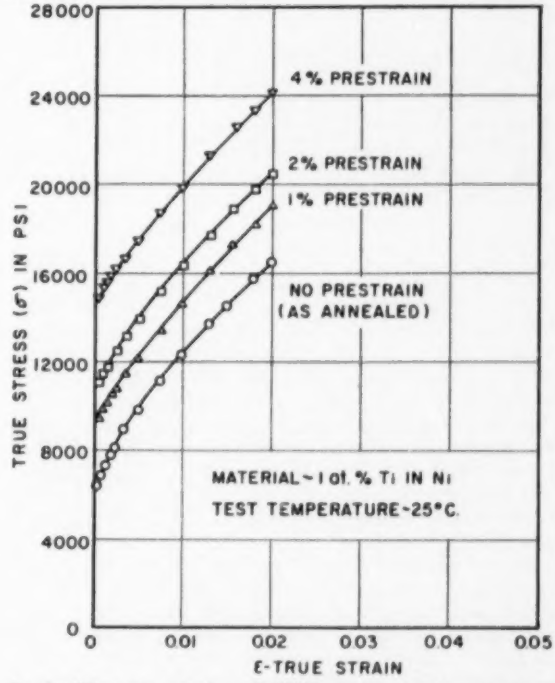


Fig. 6—Effect of prestrain and 800°C recovery treatment on tensile properties of Ni-Ti alloy.

been bent with a uniform curvature require heating to develop a substructure,[1] but other regions may form substructure boundaries even when the deformation is induced at a temperature as low as that of liquid nitrogen. The condition necessary for low temperature boundary formation is that there must be a place in the crystallite where the shear stress decreases abruptly to a stress lower than that required to keep dislocations moving. In a polycrystalline aggregate, the topology is such that there are many regions where the shear stress does decrease abruptly; substructure boundaries form in these regions during the deformation. In other regions where the stress distribution is such that bending causes a region in the crystallite to assume a uniform curvature, the dislocations remain distributed. These dislocations are stopped in the crystallites wherever they encounter internal barriers; such barriers have been shown to be effective impediments to slip.[8] Sessile dislocations are one type of internal barriers; they may be formed by the interaction of moving dislocations on two active slip systems. Quantitative data showing the hardening caused by the interaction between dislocations with different Burgers vectors which form immobile dislocations have been obtained recently for zinc single crystals tested in simple shear.[4]

In the present investigation, it was observed that the quantity of substructure boundaries developed by room-temperature deformation, i.e., with no recovery, was about the same as the number observed after an 800°C treatment. However, the nature of the small-angle boundary network is dependent upon the recovery temperature. The higher the temperatures are, the sharper the subboundaries, as is illustrated by the X-ray micrographs of Fig. 12. An example of the very sharp subboundaries formed by an 1150°C anneal is shown in Fig. 12a. Similarly, the diffraction pattern from a grain in a specimen deformed 2 pct at room temperature and recovered at 800°C is pictured in Fig. 12b. In contrast, the substructure developed in a duplicate specimen which also was strained 2 pct, but not recovered, has diffuse subboundaries as may be seen in Fig. 12c. In this case, there are three major subgrains separated by regions of severely deformed matrix; the matrix, however, is not so distorted as to prevent some of

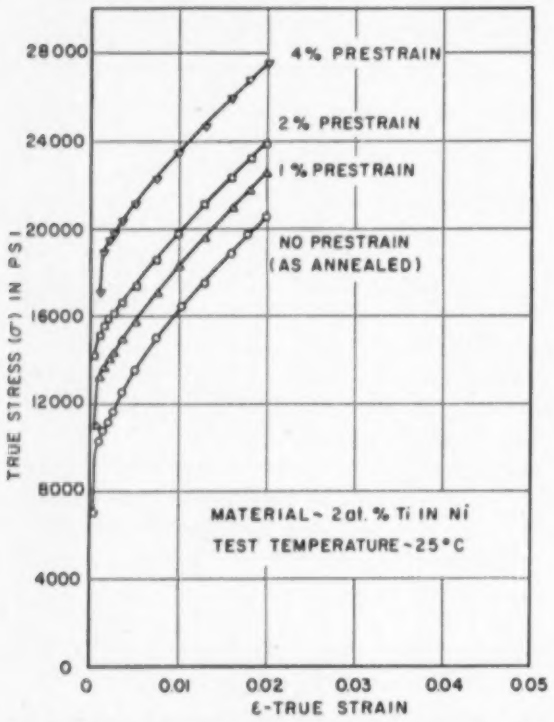


Fig. 7—Effect of prestrain and 800°C recovery treatment on tensile properties of Ni-Ti alloy.

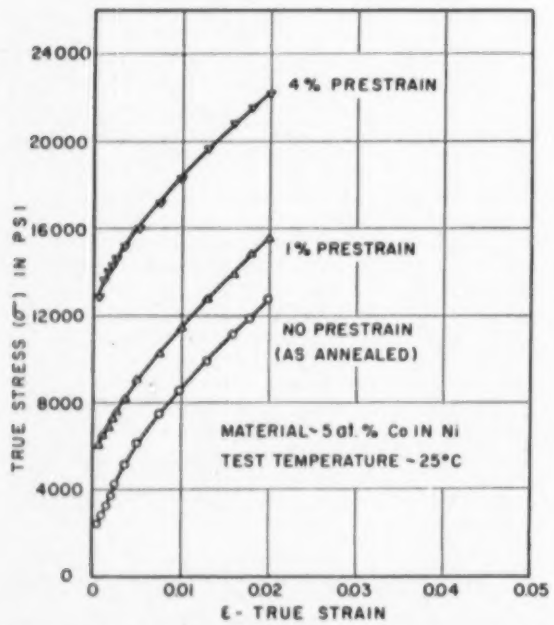


Fig. 8—Effect of prestrain and 800°C recovery treatment on tensile properties of Ni-Co alloy.

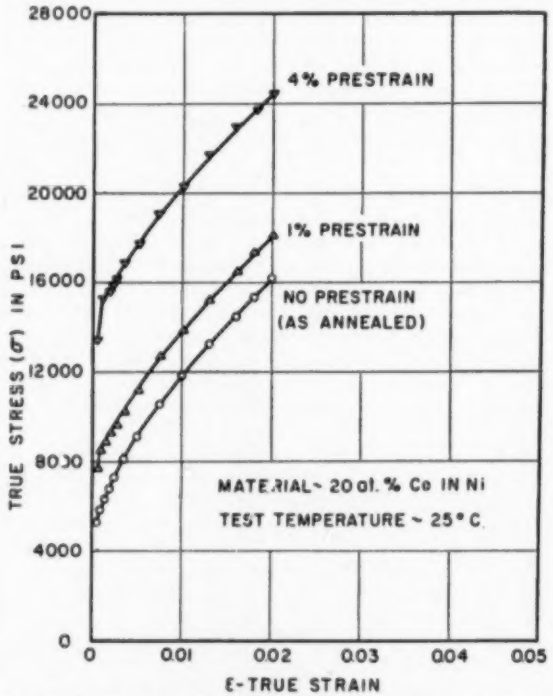


Fig. 9—Effect of prestrain and 800°C recovery treatment on tensile properties of Ni-Co alloy.

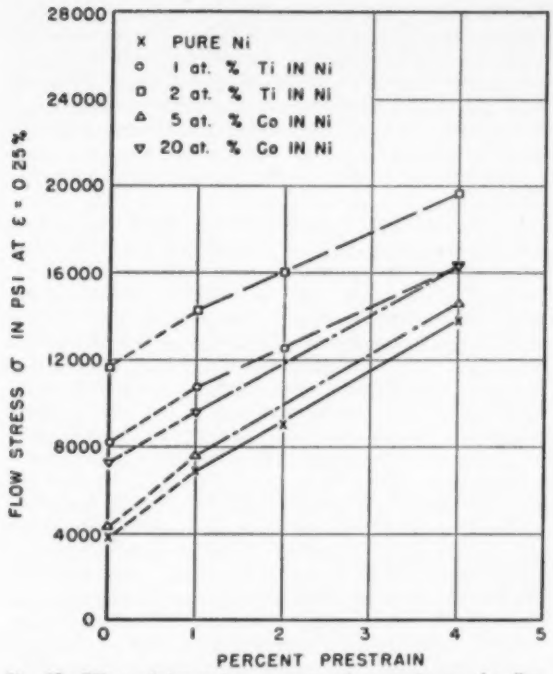


Fig. 10—Effect of increasing amounts of prestrain on the flow stress of nickel alloys.

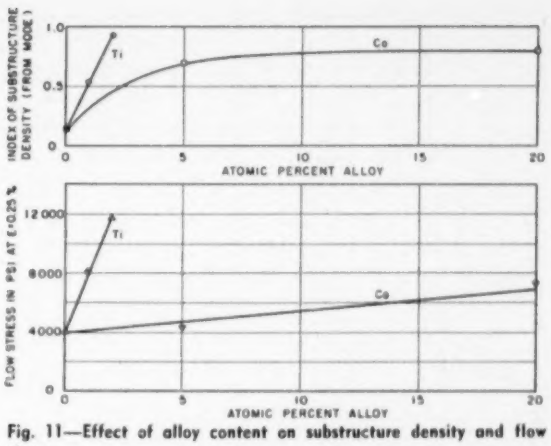


Fig. 11—Effect of alloy content on substructure density and flow stress.

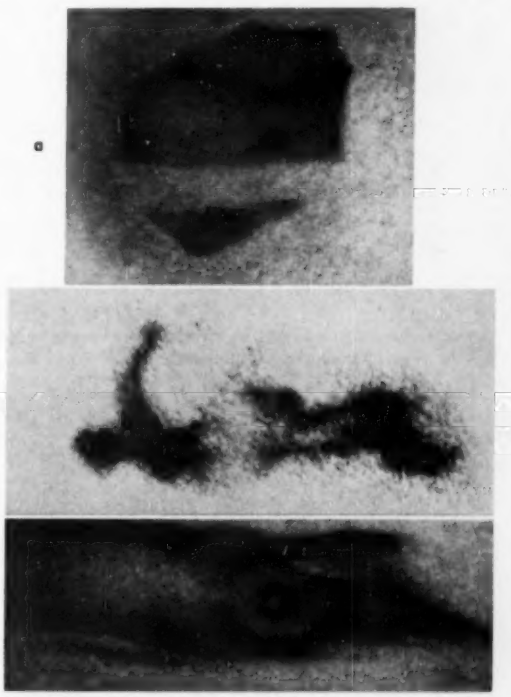


Fig. 12—X-ray micrographs of subboundaries in a pure nickel grain following deformation and recovery at various temperatures. a—1150°C, b—800°C, and c—Room temperature.

it from supporting X-ray diffraction. This type of diffraction pattern indicates that the matrix contains some general lattice bending. During recovery the distributed dislocations collect in subboundaries, a configuration which minimizes the free energy of the system.

In a current investigation, Washburn[5] has shown that the interaction of dislocations and small-angle boundaries in nearly perfect single crystals is such that unrecovered boundaries, comparable to those yielding the reflections shown in Fig. 12c, are much less effective in strengthening the metal than the clearly defined ones produced by a high temperature recovery treatment. Thus there are two general mechanisms of hardening which operate during straining or during recovery. When plastic flow is taking place, dislocations are impeded in their motion through the lattice by sessile dislocations and by other internal barriers. Such hardening is very effective, e.g., when pure nickel is strained 1 pct (Fig. 5), the strength is increased by about 6500 psi. Recovery, on the other hand, causes many of the internal barriers to disappear,[4] thus weakening the metal. During this softening process, however, the boundaries become sharper and hence more effective as barriers to moving dislocations.[6] Thus, after deformation and recovery, the metal was harder than it was in its initial state (in the authors' example by approximately 3000 psi) because of the presence of many sharp substructure boundaries. However, the metal was softer than it was after the deformation and before recovery (by approximately 3500 psi) because annealing removed many distributed dislocations trapped at internal barriers.

The present investigation was concerned with determining the effect of boundaries (developed during recovery) on the yield strength. As shown in Fig. 13, the correlation between strength and substructure density is similar for pure nickel and the Ni-Ti alloys, but the Ni-Co alloys behave in an entirely different manner. The strength of the cobalt alloys is increased markedly by prestraining and recovery, but its substructure density remains substantially unchanged. It must be concluded, therefore, that density of sharp subboundaries is not the only factor in establishing the strength of a metal.

The increase in strength in the cobalt alloys obviously was not due to an increase in the number of subboundaries. What, then, caused the strengthening? In order to understand the behavior of the Ni-Co alloys, the nature of the dislocations in the deformed metals must be considered in detail. Dislocations in the close-packed crystals are dissociated because the free energy is thereby lowered. Each dislocation becomes a pair of parallel Shockley partials.[8] The pair separate because their elastic-stress fields are mutually repulsive. Between them is a strip of fault which is hexagonal in the face-

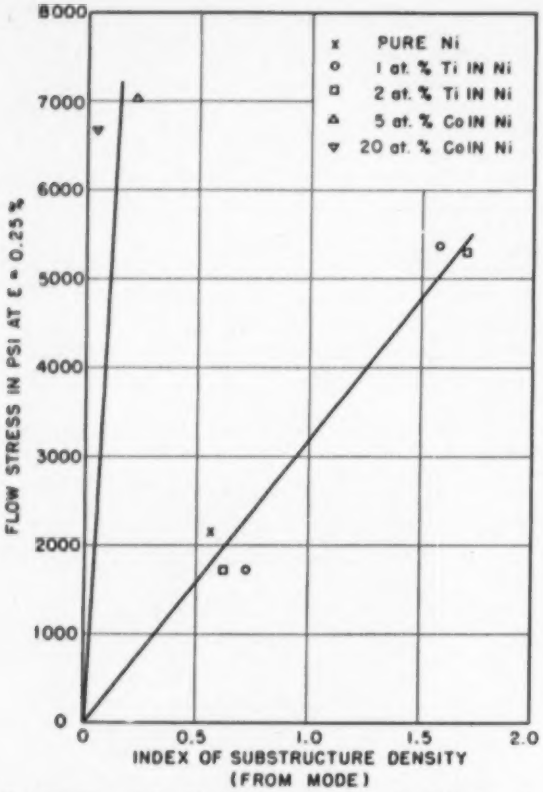


Fig. 13—The relationship between strength and substructure for various alloys of comparable initial state.

centered-cubic lattice. The generation of this faulted surface requires energy, and so the two partials cannot move apart indefinitely. The equilibrium distance between partials in an extended dislocation is determined by a balance of the force of repulsion between the partials and the surface tension of the connecting fault. The width of an extended dislocation in nickel is probably about 20 atom distances. The interaction of these extended dislocations and the solute atoms will now be considered.

The Suzuki[7] mechanism of solid-solution hardening[8] is based on the heterogeneous distribution of solute atoms in lattices containing extended dislocations. Suzuki[7] has shown by thermodynamic calculations that the concentration of solute atoms in the faulted regions, in certain cases, is higher than in the rest of the lattice. Since the cobalt atom is very nearly the same size as the nickel atom and prefers the hexagonal configuration, it is probable that, at equilibrium, the concentration of cobalt in the extended dislocations is much higher than in the rest of the lattice. The faulted regions, with their high concentration of foreign atoms, act as barriers to impinging dislocations and therefore produce hardening. Partial dislocations must be forced free from their pinning atmospheres before plastic flow can proceed and this requires high shear stresses. The free energy of extended dislocations is so lowered by the collection of some foreign atoms that even at high temperatures such dislocations are not readily removed from the lattice, nor can they migrate easily to form small-angle boundaries. The distributed dislocations would, therefore, tend to remain distributed during the recovery treatment, and would not collect to form new boundaries. Deformation always increases the dislocation density but it does not necessarily always increase the substructure density; hence, a metal may gain in strength because of dislocations distributed through the volume of a crystal, which are anchored by the Suzuki mechanism, even though there is no appreciable change in the density of sharp subboundaries (Fig. 13).

Partial dislocations are never pure edge type; nevertheless they are associated always with compressed and expanded regions of a lattice. The presence of a titanium atom in a nickel lattice causes high hydrostatic stresses because it is about 17 pct larger than the nickel. Since an accumulation of the titanium atoms in the expanded portions of the partials will reduce these stresses, it seems likely that Cottrell[9] atmospheres form in the Ni-Ti alloys. However, the number of atoms involved necessarily is much smaller than that required for a Suzuki atmosphere. Thus it is expected that the partials in the alloys containing titanium could be freed much more readily from their atmospheres than the partials in the Ni-Co alloys. The atmospheres in the titanium-bearing alloys apparently are no more effective in hindering dislocation migration into boundaries than are the impurity atoms in the "pure" nickel. This is evident from the fact that the pure nickel and Ni-Ti alloys have the same strength vs small-angle boundary density characteristics (Fig. 13).

Summary and Conclusions

1—Increasing amounts of deformation followed by recovery caused a regular gain in the strength of pure nickel, Ni-Ti, and Ni-Co alloys. The recovered metals were not as hard as the unrecovered ones because many internal barriers to slip were annihilated during recovery. In contrast to this softening effect, the small-angle boundaries introduced by the room-temperature deformation become sharper during recovery and thus become much more effective in hardening the metal.

2—The strength of the annealed alloys increased linearly with increasing alloy content.

3—The substructure density of the alloys rose with increasing titanium content but approached a maximum with increasing quantities of cobalt. For pure nickel and for the Ni-Ti alloys, the small-angle boundary density also increased linearly with increasing amounts of room-temperature deformation. For the cobalt alloys, however, the substructure density approached a maximum. Therefore, a substructure density is not the only factor in establishing the strength.

4—The number of small-angle boundaries developed by room-temperature deformation is substantially unchanged by heating up to temperatures as high as 800°C. The nature of the network, however, is dependent on the recovery temperature; the higher the temperatures, the sharper the subboundaries will be.

5—Dislocation density may increase without a corresponding increase in substructure density. This was found to be the case for the Ni-Co alloys. Hardening in these alloys may involve the Suzuki mechanism, with the extended dislocations being immobilized by a cobalt-atom atmosphere even at high temperatures. In such a case, subboundaries would not be developed in proportion to the increase in dislocation density.

6—The Cottrell hardening mechanism may be

operative in the Ni-Ti alloys, since the titanium atom is nearly one-fifth larger than the nickel atom. If it is, the titanium atmosphere is relatively ineffective in altering the recovery process, since both the pure nickel (containing minor impurities) and the titanium alloys behaved in the same way during recovery.

Acknowledgment

The authors wish to thank the United States Atomic Energy Commission for sponsoring this work. We are particularly grateful to D. W. Lillie for his encouragement and support. We are indebted to Prof. Elizabeth L. Scott and J. R. Blum of the Statistical Laboratory, University of California, Berkeley, for suggesting the Wilcoxon test as a means of analyzing the substructure data, for devising the necessary modification, and for helpful discussion. We also wish to express our sincere appreciation to T. H. Hazlett without whose help this work could not have been conducted.

Appendix A: A Modification of the Wilcoxon Test

Application of the Wilcoxon test tests the hypothesis that two samples come from the same distribution.

If $X_1, \ldots, X_n$ is a sample from a distribution F, and $Y_1, \ldots, Y_m$ is a sample from a distribution G, the hypothesis to be tested is that $F = G$. Since there are ties present, i.e., X_i may equal X_j for $i = j$ and X_i may equal Y_k for any i and k, a modification of the usual Wilcoxon test was devised.

The test procedure is as follows: The two samples are combined into one which is arranged in increasing magnitude. Let r_i, $i = 1, \ldots, n$, be the rank of the ith smallest X in the combined ordered sample and similarly let the s_j, $j = 1, \ldots, m$, be the ranks of the Y's. The sum of the ranks of the Y's is computed $S = \sum_{j=1}^{m} s_j$. As a check, the sum of the ranks of the X's are also computed $R = \sum_{i=1}^{n} r_i$, since $S + R$ is the sum of all the ranks, i.e., $S + R = 1 + 2 + \ldots + (m + n) = (m + n)(m + n + 1)/2$.

In any case where ties occur, the contribution to the sum of the ranks rather than the individual rank is computed as follows: If there are iX's and jY's tied for a position in the combined-order sample, then the starting position for these will be $1 + v$ when v equals the total number of X's and Y's which precede the iX's and jY's. The last position is $v + i + j$, and therefore the midrank is $(2v + i + j + 1)/2$. Then the contribution to the sum of the ranks of the X's is $C_x = i(2v + i + j + 1)/2$ and the contribution to the sum of the ranks of the Y's is $C_y = j(2v + i + j + 1)/2$.

Next, the expected value of S, $E\{S\}$, and the variance of S, σ_s^2, are computed. When the hypothesis is true

$$E\{S\} = \frac{m}{2}(m + n + 1)$$

and

$$\sigma_s^2 = \frac{m \cdot n}{12}(m + n + 1).$$

Now let $T = S - E\{S\}/\sigma_s$; then if m and n are moderately large (say > 10) and if the hypothesis is true, T is approximately normally distributed with mean zero and variance one. Hence T should be expected to fall well within the normal range most of the time.

Assume the value of T computed to T_0. In order to compute the probability of finding a value of T as large in absolute value as T_0 (from the normal tables) the value of $p = 1 - \int_{-|T_0|}^{|T_0|} \exp(-u^2/2)\,du$ is found. If this probability p is small (e.g., <0.05), the hypothesis would be rejected and an error in doing so would be made only on the average of 5 pct of the time. If the probability is large (e.g., >0.05), the hypothesis is accepted.

As an example the following test is included here: Hypothesis: the substructure boundary-distribution curves for pure Ni and 1 atomic pct Ti in Ni at $\epsilon = 0$ pct come from the same sample.

X_i (Ni)	Y_j (1 Atomic Pct Ti in Ni)	v	$(2v+j+i+1)/2$	C_x	C_y
87	93	0	90.5	7873.5	8416.5
13	53	180	213.5	2775.5	11315.5
1	17	246	255.5	255.5	4343.5
1	7	264	268.5	268.5	1879.5
—	3	272	274.0		822.0
$n = 102$	1	275	276.0		276.0
	0	276	276.5		0.0
	1	276	277.0		277.0
	$m = 175$			$R = 11173.0$	$S = 27330.0$

$$R + S = 38503.0$$

$$\frac{(n+m)(n+m+1)}{2} = \frac{(102+175)(102+175+1)}{2} = 38503.0$$

$$E(S) = \frac{175}{2}(278) = 24325; \quad \sigma_s^2 = \frac{(102)(178)}{12}(278) = 413525$$

$$\sigma_s = 643 \qquad T = \frac{27330 - 24325}{643} = 4.67$$

$$p = 1 - 0.999^{+} = 0$$

Hence the probability of obtaining a value of $|T|$ as large as $|4.67|$ is very nearly zero; therefore the X_i and Y_j come from different distributions.

Appendix B: Transformed Cumulative Frequency Curves (Figs. 14 through 18)

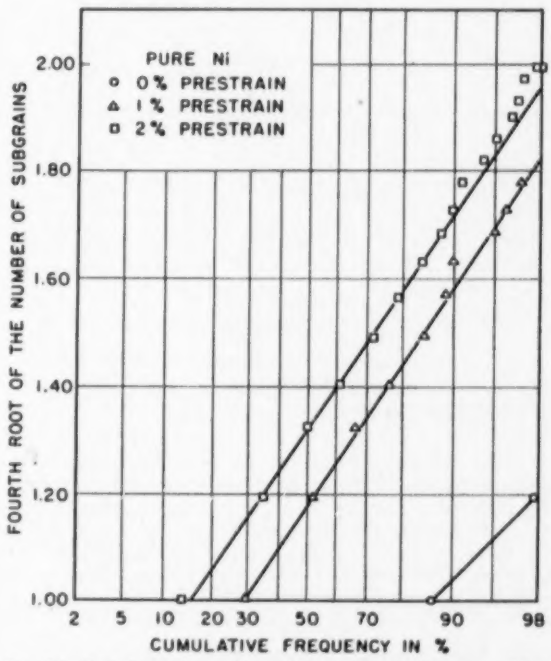


Fig. 14—Transformed cumulative frequency plot for pure nickel at various prestrains.

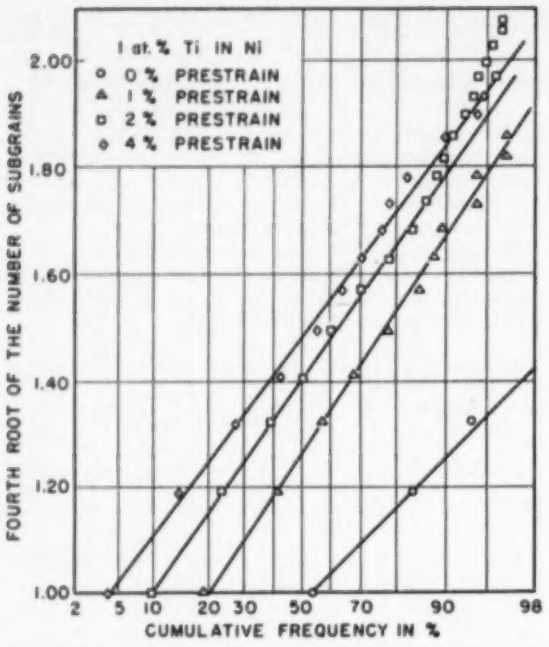

1 at. % Ti IN Ni
0 % PRESTRAIN
1 % PRESTRAIN
2 % PRESTRAIN
4 % PRESTRAIN
FOURTH ROOT OF THE NUMBER OF SUBGRAINS
CUMULATIVE FREQUENCY IN %

Fig. 15—Transformed cumulative frequency plot for 1 atomic pct Ti in nickel for various prestrains.

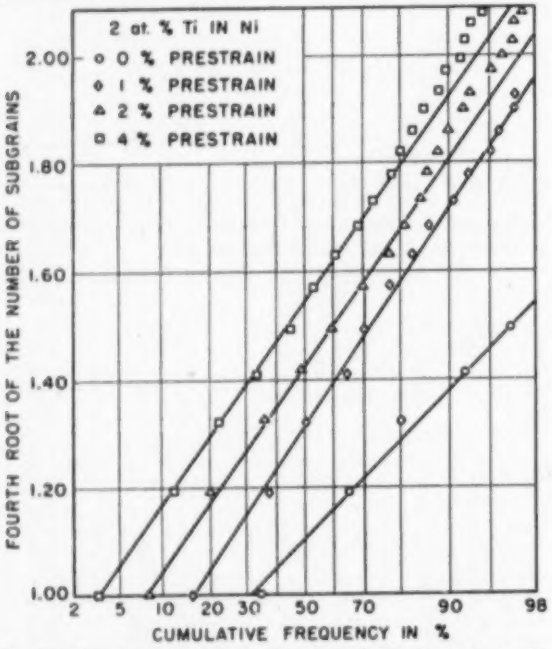

2 at. % Ti IN Ni
0 % PRESTRAIN
1 % PRESTRAIN
2 % PRESTRAIN
4 % PRESTRAIN
FOURTH ROOT OF THE NUMBER OF SUBGRAINS
CUMULATIVE FREQUENCY IN %

Fig. 16—Transformed cumulative frequency plot for 2 atomic pct Ti in nickel for various prestrains.

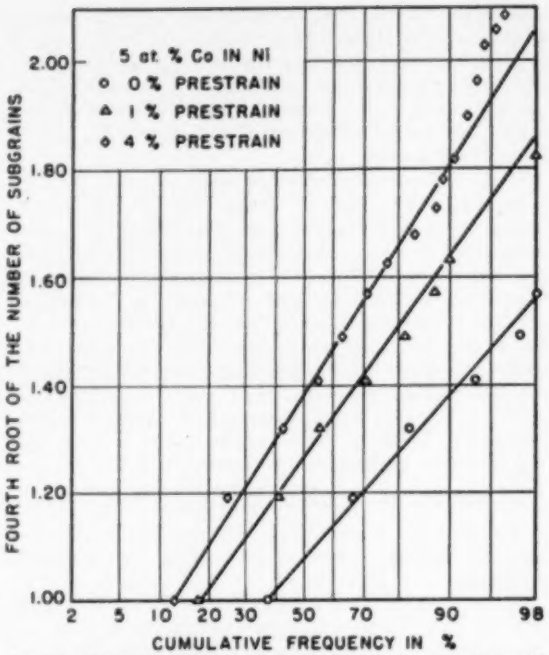


Fig. 17—Transformed cumulative frequency plot for 5 atomic pct Co in nickel for various prestrains.

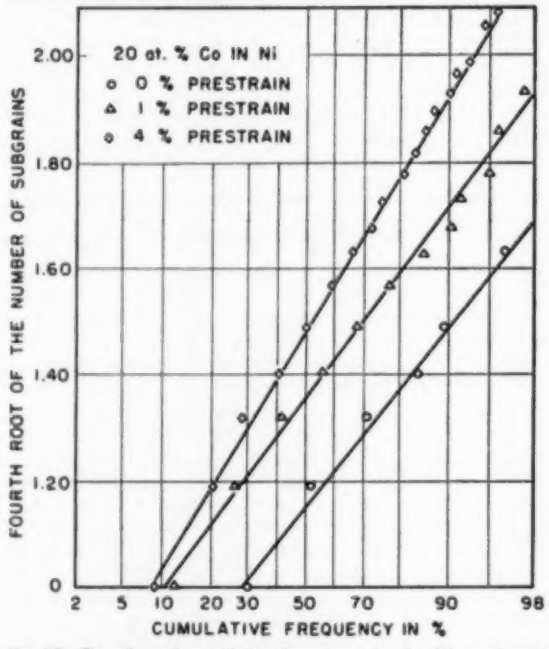


Fig. 18—Transformed cumulative frequency plot for 20 atomic pct Co in nickel for various prestrains.

References

[1] R. W. Cahn: *Journal* Institute of Metals (1951) **79**, pp. 129-158.

[2] C. S. Barrett: *Trans.* AIME (1945) **161**, pp. 15-64.

[3] E. H. Edwards, J. Washburn, and E. R. Parker: *Trans.* AIME (1953) **197**, pp. 1525-1529; JOURNAL OF METALS (November 1953).

[4] E. H. Edwards and J. Washburn: To be published by AIME.

[5] J. Washburn: Private communication.

[6] W. T. Read, Jr.: *Dislocations in Crystals.* (1953) McGraw-Hill Book Co.

[7] H. Suzuki: Chemical Interaction of Solute Atoms with Dislocations. *Science Reports of the Research Institute* Tahaku University, Japan (1952) **A4**, No. 5, pp. 455-463.

[8] E. R. Parker and T. H. Hazlett: Principles of Solution Hardening. *Relation of Properties to Microstructure.* To be published by ASM.

[9] A. H. Cottrell: Effect of Solute Atoms on the Behavior of Dislocations. *Report on Conference on Strength of Solids* Physical Society, London (1948) pp. 30-38.

AIME OFFICERS:
President—Leo F. Reinartz
Vice-Presidents—T. B. Counselman, L. E. Elkins, Harold Decker, O. B. J. Fraser, A. B. Kinzel, J. B. Morrow
President-Elect—H. DeWitt Smith
Treasurer—G. F. Moulton
AIME STAFF:
Secretary—E. H. Robie
Ass't. Secretaries—J. B. Alford, H. N. Appleton, E. J. Kennedy, Jr., Ernest Kirkendall, R. E. O'Brien
Ass't. Treasurer—P. J. Apol

Annual Fall Conference, Southern Ohio Section, NOHC To Be Held in Columbus, Ohio

THE Deshler-Hilton Hotel in Columbus, Ohio, will be the scene of the November 5 to 6 meeting of the Southern Ohio Section, National Open Hearth Conference. Registration will be at 9:00 am in the Ballroom. The Rev. Lawrence Hall of St. Paul Episcopal Church will be the speaker at the dinner, 7:00 pm on Friday. Toastmaster for the dinner will be Lee Lambing, steelmaking consultant.

FRIDAY, NOVEMBER 5

9:00 am, Ballroom

REGISTRATION

10:30 am to 12:30 pm, Ballroom

1. Welcoming Remarks:
   H. P. Gaw, Works Metallurgist, Armco Steel Corp., Middletown, Ohio, and Chairman, Southern Ohio Section, National Open Hearth Committee.
2. Southern Ohio Section Award Paper

OPERATING AND COMBUSTION SESSION

Chairmen:

C. F. Staley, Superintendent No. 2 Open Hearth, Armco Steel Corp., Middletown, Ohio
R. Thornberry, Superintendent, Open Hearth, Newport Steel Corp., Newport, Ky.

1. **Effect of Blast Furnace Analyses on Open Hearth Production and Preliminary Analyses:**
   G. B. Reisenberg, Metallurgist, Armco Steel Corp., Middletown, Ohio
2. **Instrumentation on New Furnaces at Detroit Steel Corp., Portsmouth, Ohio Works:**
   Frank McGough, Superintendent of Fuel and Power, Detroit Steel Corp., Portsmouth, Ohio.
3. **Maintaining Open Hearth Performance with Multiple Fuels:**
   Robert Sigl, Fuel Observer, Armco Steel Corp., Middletown, Ohio.

1:00 to 2:00 pm, Ballroom

LUNCHEON

2:30 to 5:30 pm, Ballroom

PRACTICE AND QUALITY SESSION

Chairmen:

Albert Meihack, Superintendent Steel Production, Empire Steel Corp., Mansfield, Ohio
Charles R. Taylor, Supervising Metallurgist, Armco Steel Corp., Middletown, Ohio

1. **Use of Motion Pictures in the Study of Steel Pouring Practices:**
   A. B. Blockridge, Jr., Metallurgical Engineer, Jones & Laughlin Steel Corp., Research Laboratories, Pittsburgh
2. **Results Obtained with Refract-All Gun:**
   Edward Reyer, Superintendent, Open Hearth, International Harvester Co., Wisconsin Steel Div., Chicago, Ill.
3. **Recent Developments in Mold and Stool Design:**
   George Dorin, Engineer, Shenango Penn Mold Co., Sharpsville, Pa.
4. **Measurement of Bath Temperature by Immersion Thermocouples:**
   Frank Williams, Senior Metallurgist, Armco Steel Corp., Ashland, Ky.

7:00 pm, Ballroom

DINNER

Toastmaster:

Lee Lambing, Steelmaking Consultant, and Chairman, National Open Hearth Steel Committee

Speaker:

Rev. Lawrence Hall, St. Paul Episcopal Church, Cleveland, Ohio:

Life With a Capital L

SATURDAY, NOVEMBER 6

9:00 am, Room 216

National Open Hearth Executive Committee Meeting

NEW COST-SAVING PROCESS FOR FOUNDRIES

The Airco Carbide Injection Process in use at the American Brake Shoe Company, Mahwah, N. J.

Carbide injection method developed by AIRCO® cuts ductile iron alloy costs up to 50 percent

permits melting in acid cupolas

Many foundries that have limited their production to gray iron will soon enter the ductile iron field, thanks to a new Airco development. Desulfurization of molten iron by injecting calcium carbide can reduce sulfur content to such levels that ductile iron can be melted in ordinary acid cupolas. At the same time, this sulfur content — as low as 0.01 percent — makes it possible to cut the amount of expensive alloy elements drastically with no sacrifice of mechanical properties. Foundries already using Airco carbide injection report savings of up to 50 percent on these alloy costs alone!

The injection equipment itself is simple and inexpensive. It consists of a hopper and an injection tube through which the powdered carbide is injected into the molten iron by a stream of nitrogen. The operating expenses are the cost of the gas and the carbide, trivial in comparison to the savings they make possible.

Calcium carbide injection is a fruitful result of Airco Technical Service's constant effort to find new profitable techniques for customers. If you have a welding, cutting or metallurgical problem, why not let Airco help you develop the most practical approach to it? It can pay big dividends.

AIR REDUCTION

60 East 42nd Street • New York 17, N. Y.

at the frontiers of progress you'll find...

AN AIR REDUCTION PRODUCT

Divisions of Air Reduction Company, Incorporated, with offices and dealers in most principal cities

Air Reduction Sales Company
Air Reduction Pacific Company

Represented Internationally by
Airco Company International

Foreign Subsidiaries: Air Reduction Canada Limited, Cuban Air Products Corporation

1954 AIME Budget Reviewed

The AIME budget for 1954 was carefully reviewed at the July 26 meeting of the Executive and Finance Committees in the light of actual results for the first six months of the year. No change was thought necessary in the overall items for Income except that for advertising, which was reduced from $218,000 to $217,000. In the Expense category, Technical Divisions was raised from $68,500 to $69,000; Special Membership Activity from $7000 to $9000; cost of Journals from $310,300 to $314,300; and cost of Transactions Supplement reduced from $18,000 to $13,000. The Income budget does not include any money to be raised outside for publishing metals research papers, which will remain segregated until a special appropriation for its use is voted. The revised budget indicates a deficit for the year of $1300.

A. A. Winne Awarded Fritz Medal

The John Fritz Medal for notable scientific or industrial achievement has been awarded to Harry Alonzo Winne. Until his retirement last November, Mr. Winne was vice president in charge of engineering for General Electric Co., with which his entire professional career was associated. His citation reads: "Engineer, industrial statesman, for service to his country in war and peace through his distinguished leadership in the electrical industry." Mr. Winne is a member of the ASME and AIEE.

Section Delegates' Travel To Be Paid

As in past years, the AIME will pay the round trip first class rail and roomette cost of travel of Section Delegates to the Annual Meeting in Chicago next February. If travel is by air or other means, the amount of the rail fare will be allowed. The amount and formula for other financial support to Local Sections will be determined by the Board of Directors at the Annual Meeting.

A. P. Sloan, Jr. Named Hoover Medalist

Alfred P. Sloan, Jr., chairman of the board of the General Motors Corp., has been named Hoover Medalist for 1954, the 15th recipient. He is a member of the Society of Automotive Engineers. Presentation of the medal will be made at the annual dinner of the American Institute of Consulting Engineers in the Keystone Room of the Hotel Statler, New York, on October 19. Herbert Hoover will attend. The medal is awarded for distinguished public service by an engineer.

Southwestern NOHC To Meet in Atlanta, Ga.

The Southwest Section of the AIME National Open Hearth Committee will meet at the Henry Grady Hotel, Atlanta, Ga., from October 7 to 9.

A trip through the Atlantic Steel Co. plant followed by a barbecue at the plant recreation park will open the program October 7. Friday, October 8, will be given over to technical meetings. On the agenda are papers on open hearth production at Monterey, progress in continuous casting, casting of small ingots, European steelmaking practices, use of electronic reporting spectrograph, and operations of electric furnaces including a comparison with open hearths.

The program closes on Saturday, October 9, with members attending the LSU vs Georgia Tech football game.

"Porphyry Coppers" Revision Planned

A. B. Parsons, former Secretary of the AIME and author of *The Porphyry Coppers*, published in 1933, is now bringing the book up to date. As before, the Rocky Mountain Income Fund is underwriting the volume, and the Executive and Finance Committees of the Institute have approved an expenditure of $7500 for the purpose. A book of some 180 pages is contemplated. The history of the 12 properties covered in the earlier book will be brought up to date, and the newly developed properties in North and South America will be treated in much the same way as before. Engineering and technologic progress of the last quarter century as it relates to the porphyries will be summarized.

Local Section Transfers Approved

At its meeting on July 26, the AIME Executive Committee approved release of Moore and Hartley Counties, Texas, from the Panhandle to the Hugoton Local Section. Carson County, Texas, was added to the area of the Panhandle Section. A request for recognition of the New York Petroleum Section was approved, subject to agreement by the executive committee of the New York Section and the working out of arrangements involving its cooperative functions. The new Section would include the five counties comprising New York City (New York, Bronx, Kings, Queens, and Richmond); Nassau and Suffolk Counties on Long Island; Rockland, Orange, Putnam, and Westchester Counties in New York; and Bergen, Essex, Hudson, Middlesex, Monmouth, Passaic, Morris, Somerset, Sussex, and Union Counties in New Jersey.

European Meeting Plans Take Shape

The Joint Metallurgical Societies meeting in Europe is beginning to shape up as one of the most significant technical gatherings in many years from the point of view of science and promotion of free interchange of knowledge.

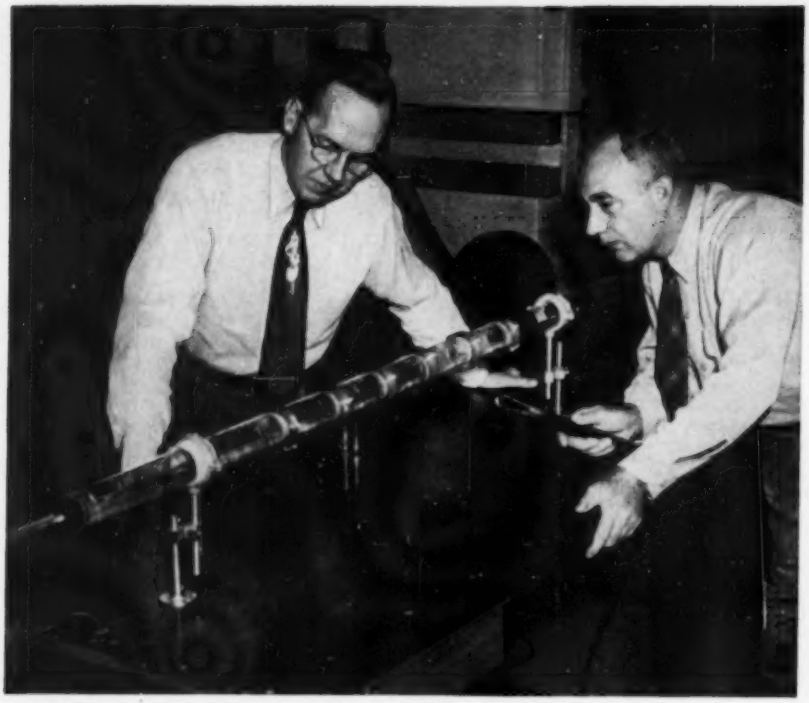
The Joint Committee of AIME and ASM charged with smoothing the path of delegates from this side of the Atlantic held its first meeting in New York Sept. 9. The committee's task is to encourage individuals to attend the European meetings and also to encourage companies to make it possible for their personnel to make the trip.

U. S. metallurgists who take part in the affair will be representative of their profession in this country, rather than delegates of individual societies. Another job facing the committee is the selection of the most qualified persons in the event that more than the 300 planned for wish to go.

Travel arrangements may be made through Thomas Cook & Sons. The program of the meetings can be obtained by writing to the Secretary of the Metals Branch, AIME.

Raise Funds To Publish Metals Research Papers

Following authority given at the March 17 meeting of the Executive and Finance Committees of the Institute, efforts have been made to raise a fund of $50,000 for the publication of Technical Publications reporting results of metals research. Metals Branch deficits in recent years have largely resulted from publication of a large amount of material of this kind, and it is hoped that funds thus received, which will be solicited annually, will make possible a balanced budget for the Branch. Industrial firms interested in, or benefiting from, publication of the results of research in the metals producing industry are the ones being approached. By September 9, $18,000 had been received by the Institute. E. O. Kirkendall is directing the Metals Branch Research Publication Fund activities.



Zone Refining apparatus, showing tube and induction-heating coils. For transistors—tiny electronic amplifiers—germanium is made extremely pure. Then special impurities are added in controlled amounts for best transistor performance.

1 part in 10,000,000,000

To make the most of their revolutionary invention, the transistor, Bell Laboratories scientists needed ultra-pure germanium.

The scientists solved their problem by devising a radically new refining process. The germanium it yields may well be the purest commercially produced material on earth.

It has only *one part in ten billion* of impurities harmful to transistor performance. That's about the same as a pinch of salt in 35 freight cars of sugar.

Yet the new process, Zone Refining, is simple in principle. An ingot of germanium is drawn through a series of induction-heating coils that melt narrow zones of the substance. Since impurities are more soluble in the liquid than in the solid form of a metal, the molten zones collect impurities. They are swept along by the successive melts to the end of the ingot, which is finally cut off.

Zone Refining is also being applied to the ultra-purification of other materials useful to telephony. This single achievement of research at Bell Telephone Laboratories clears the way for many advances in America's telephone system.

BELL TELEPHONE LABORATORIES

IMPROVING AMERICA'S TELEPHONE SERVICE PROVIDES CAREERS
FOR CREATIVE MEN IN SCIENTIFIC AND TECHNICAL FIELDS

Personals

W. T. Bradley, International Minerals & Chemical Corp., is now located at the Chicago offices as assistant to the general manager, phosphate minerals div. He had been staff metallurgist of the Florida phosphate operations.

J. FRENCK

John Frenck has joined the metals research laboratories of the Electro Metallurgical Co., Niagara Falls, N. Y., as technical assistant. He gained technical experience while employed by the Belle City Malleable Iron Co. He also held a research assistantship at the University of Wisconsin.

Frank B. Macron is now employed as plant engineer for the Alpha Portland Cement Co., Cementon, N. Y. He was previously associated with the Hudson Coal Co., Scranton, Pa.

Keki K. Irani, formerly associated with Algoma Steel Corp., Sault Ste. Marie, Ontario, is now with the Canadian Allis-Chalmers, Lac Line, Quebec.

A. S. Contrini is metallurgist and plant manager for Zinc Nacional S. A., Monterrey, Mexico.

George J. Fischer was appointed director of the metallurgical dept. of Sam Tour & Co., Inc., New York. Mr. Fischer has been a metallurgist for the Western Electric Co. and an instructor at the Polytechnic Institute of Brooklyn.

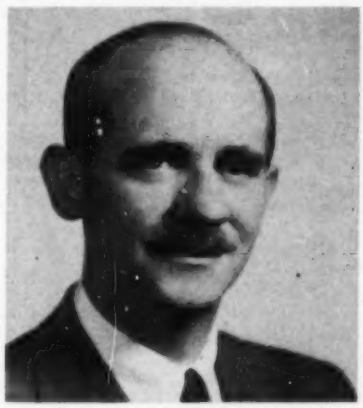
James B. Austin, Max W. Lightner, and **Robert W. Holman,** U. S. Steel Corp. have been appointed assistant vice presidents to head the activities of the fundamental research, applied research and development, and operations research groups, respectively. Dr. Austin is located at Kearny, N. J., Mr. Lightner is in Pittsburgh, and Mr. Holman will transfer from Gary, Ind., to Pittsburgh.

H. Gordon Poole, former associate professor of extractive metallurgy, Case Institute of Technology, Cleveland, has joined the staff at the Colorado School of Mines, Golden, as professor of metallurgy and head of department.

Robert H. Aborn, assistant director of the fundamental research laboratory, U. S. Steel Corp., Kearny, N. J., has been named director. Dr. Aborn has been associated with U. S. Steel for 24 years. **Lawrence S. Darken** has been named assistant director of physical chemistry; **Bernard M. Larsen,** assistant director of metallurgical processes; and **Reginald L. Rickett,** assistant director of physical metallurgy.

John N. Reding has joined Dow Chemical Co., magnesium div., as a research and development engineer for the metallurgical laboratory, Midland, Mich. Mr. Reding had been an associate chemical engineer with the Armour Research Foundation of the Illinois Institute of Technology, Chicago.

Alfred Strasser is a metallurgist for Nuclear Development Associates, White Plains, N. Y. Mr. Strasser had been a metallurgist at the Wright Patterson Air Force Base, Dayton, Ohio.

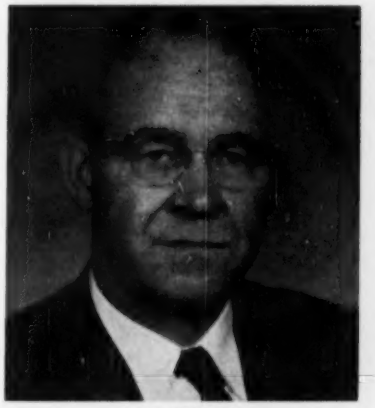


S. T. JAZWINSKI

S. T. Jazwinski has been appointed director of research for Barium Steel Corp. subsidiaries. In 1946 he had been named chief metallurgist for Barium Steel Corp., and was named vice president in charge of two subsidiaries. He was named chief research metallurgist in 1952.

Alfred Sugar has been appointed vice president and technical director of Alloys & Chemicals Mfg. Co., Cleveland. Mr. Sugar had been aluminum div. plant manager of the American Metal Co., Ltd.

Henry G. Holtz has accepted the position of metallurgical engineer, materials and processes laboratory, General Electric Co., Schenectady, N. Y. He had been chief metallurgist for Taylor Forge & Pipe Works, Chicago.



H. V. BEASLEY

H. V. Beasley has been named to head the newly established constructional alloy steels section of the International Nickel Co., development and research div., New York. **T. E. Kihlgren** is in charge of the nickel alloys development section. **V. N. Krivobok** heads the stainless steel and heat resistant alloys section. **W. H. Sparr, Jr.,** has been appointed to succeed Mr. Beasley in charge of the technical field section, Pittsburgh.

Eugene H. Kinelski has joined Dwight-Lloyd, Inc., Netcong, N. J., and will be engaged in the research, development, and sale of ore beneficiation plants, sintering and pyrolizing equipment. Prior to accepting this position, Mr. Kinelski was associated with Mellon Institute of Industrial Research, Pittsburgh.

Richard B. Brennan has joined E. I. du Pont de Nemours & Co., engineering research laboratory, experimental station, Wilmington, Del.

G. R. Fish, American Smelting & Refining Co., has been transferred from the Garfield, Utah office to New York.

Arthur F. Hallam is a physicist for the Firestone Tire & Rubber Co., Akron, Ohio. Mr. Hallam had been a metallurgist for the Clevite-Brush Development Co., Cleveland.

Raymond E. Byler has been named business manager of the western laboratories div., (formerly the Merrill Co.) Arthur D. Little, Inc., San Francisco.

Kenneth K. Tucker has joined the Newman Machine Co., Greensboro, N. C. He had been associated with the Rockwell Mfg. Co., Pittsburgh.

Warren Chernock is senior research scientist with Olin Industries, New Haven, Conn.

J. F. B. JACKSON

J. F. B. Jackson has resigned his position as director of the British Steel Castings Research Assn., Sheffield, England, and has joined the board of A. P. V.—Paramount Ltd., Sussex, England as deputy managing director.

Kenneth H. Wright has accepted a position in the ore div. engineering dept., of the Jones & Laughlin Steel Corp., New York.

Leon H. Nelson has been appointed director of conservation and yield control for Crucible Steel Co. of America, Pittsburgh. Mr. Nelson had been associated with the Republic Steel Corp. prior to joining Crucible.

Allan M. Short has returned to the U. S. after a five year stay in Saudi Arabia and is now consultant geologist and mineral technologist in Denver.

Ronald C. Koo is employed by the Weston Electrical Instrument Corp., Newark, N. J., as a metallurgical engineer.

Henry L. Tear has become superintendent of open hearth for the Washburn Wire Co., Phillipsdale, R. I. Mr. Tear had been superintendent, open hearth 4, Jones & Laughlin Steel Corp., Pittsburgh, Pa.

William C. Buell, Jr., has retired as a member of the consulting staff of Arthur G. McKee & Co., Cleveland.

Jack H. Wernick has accepted a position with the Bell Telephone Laboratories, Murray Hill, N. J.

Paul Gordon has been appointed associate professor of metallurgical engineering at the Illinois Institute of Technology, Chicago.

Robert E. Blackwood is chief consultant and ICC inspector for T. Harry Cochrane Laboratories, Milwaukee. Mr. Blackwood had been process engineer for A.C. Spark Plug, Flint, Mich.

D. W. Lillie, chief of the metallurgy and materials branch, div. of research, U. S. Atomic Energy Commission, Washington, D. C. has joined the research laboratory of the General Electric Co., Schenectady, N. Y.

Donald L. Byrne is now employed as a junior metallurgist with the research dept. of the Allegheny Ludlum Steel Corp., Dunkirk, N. Y.

Curtis L. Wilson, dean of the Missouri School of Mines and Metallurgy, Rolla, Mo., was recently awarded the honorary degree of Doctor of Engineering from the Washington University, St. Louis.

J. D. Dickerson has been named chief metallurgist, Midland Works, Crucible Steel Co. of America. Prior to joining Crucible in 1953 Dr. Dickerson was chief metallurgist for the Republic Steel Corp., Buffalo, N. Y.

Russell T. Drennan has been named general sales manager of the chemicals div., Kaiser Aluminum & Chemical Corp., Oakland, Calif. **John Minshall** has been assigned as eastern regional sales manager for the chemical div., Akron, Ohio. Mr. Drennan joined Kaiser Chemicals in 1946 as chief chemist of the Baton Rouge, La., alumina plant. Mr. Minshall has been associated with the firm since 1941.

Harold D. Kessler is supervisor of the metallurgical div., Titanium Metals Corp. of America, Henderson, Nev. Mr. Kessler had been associated with the Armour Research Foundation of the Illinois Institute of Technology, Chicago, Ill.

William A. Koppi has joined the Lebanon Steel Foundry, Lebanon, Pa. He had been connected with the Buick Motor Div., General Motors Corp., Willow Springs, Ill.

Luis Giannattasio, civil engineer of Montevideo, Uruguay and president of the Pan American Union of Engineering Societies (UPADI), has been elected to honorary membership in the ASME.

J. H. Baldrey, Electro Metallurgical Co., has been transferred from the New York office to Chicago.

Robert W. Hernlund, metallurgical engineer, has joined the faculty of the University of the Philippines under its contract with Stanford University, sponsored by the U. S. Foreign Operations Administration. Professor Hernlund was formerly sales promotion manager of Western Machinery Co., San Francisco. He will teach specialized courses and act in an advisory capacity on engineering curriculum and special programs.

Carl R. Norlander has accepted the position of sales engineer with the Columbus Bolt & Forging Co., Chicago. Mr. Norlander had been associated with Revere Copper & Brass, Inc., Chicago.

Robert E. Keith, who recently received his Ph.D. from the University of Michigan, has joined the staff of the General Electric Research Laboratory, Schenectady, N. Y.

Williams Griffith, Jr., is superintendent of industrial engineering for the Bridgeport Brass Co., Adrian, Mich. He had previously been located at Indianapolis, Ind., for the same company.

O. H. Hugill recently resigned from the Algoma Steel Corp., Ltd., Sault Ste. Marie, Ont., Canada, to accept a position with Canadian Furnace Co., Ltd., Port Colborne, Ont., Canada.

Obituaries

James A. Kavenaugh (Member 1945) consulting metallurgical engineer, died on May 28. He was residing at Arcadia, Calif. Mr. Kavenaugh was born at Denver in 1915 and graduated in 1938 from the Colorado School of Mines. For two years following graduation he was assistant heat treating metallurgist for Carnegie-Illinois Steel Corp. He joined the Naval Aircraft Factory at Philadelphia and for two years was an assistant metallurgist. In 1942 he became a materials and process engineer for Vultee, Inc. Since 1944 he has been a consultant. During this time he was employed by George A. Starbird Co., Los Angeles and was technical advisor for Revere Copper & Brass Co., Inc., Los Angeles.

Harry A. Schwartz (Member 1919), director of research for the National Malleable & Steel Castings Co. for 34 years, died on July 25. He had been ill for several months. In 1939 Dr. Schwartz had been awarded the E. J. Fox Gold Medal by the Institute of British Foundrymen. In 1930 he was awarded the John A. Penton Gold Medal by the American Foundrymen's Assn. Born in Kentucky, Dr. Schwartz received his technical education at Rose Polytechnic Institute. He joined National Malleable Castings Co., Indianapolis in 1902 and in 1920 he was named director of research. At the time of his death he was residing at Cleveland Heights, Ohio.

At the '54 Metal Exposition—Nov. 1-5 . . .

50,000-PLUS METALS INDUSTRIES MEN

WILL LEARN NEW WAYS OF CUTTING COSTS!

Your competitors . . . thousands of 'em . . . will be on hand at this great industrial event to check closely on everything that's new and improved . . . everything that will help them build better products . . . cheaper . . . faster!

They'll come from all corners of the land . . . these 50,000-plus metals industries men . . . and they'll take away with them, when this great Metal Show ends, more ideas . . . more information and more know-how than any 'stay-at-home' executive could possibly match!

Owned and Managed by
AMERICAN SOCIETY FOR METALS
W. H. Eisenman, *Managing Director*
Chester L. Wells, *Ass't. Director*

INTERNATIONAL AMPHITHEATRE
CHICAGO NOV. 1-5

NATIONAL METAL EXPOSITION

NATIONAL METAL CONGRESS

Necrology

Date Elected	Name	Date of Death
1933	Thos. Gayleon Andrews	July 1954
1937	Max W. Ball	Aug. 28, 1954
1941	Stanley Barber	July 31, 1954
1953	Ali DeSzepessy Schaurek	May 1954
1926	William Harkes	Aug. 11, 1954
1918	James O. Lewis	June 15, 1954
1920	Keith Roberts	Mar. 16, 1954
1918	Frederick LeV. Service	July 22, 1954
1930	Harry A. Swem	Aug. 8, 1954
1920	George H. Taber, Jr.	Aug. 30, 1954
1936	Seton S. Williams	May 26, 1954
1927	Clifford S. Wilson	May 1954

Proposed for Membership

— Metals Branch AIME —

Total AIME membership on Aug. 31, 1954 was 21,327, in addition 1724 Student Associates were enrolled.

ADMISSIONS COMMITTEE

O. B. J. Fraser, Chairman; R. B. Caples, Vice-Chairman; F. A. Ayer, A. C. Brinker, R. H. Dickson, Max Gensamer, Ivan A. Given, Fred W. Hanson, T. D. Jones, Sidney Rolle, J. H. Scaff, John T. Sherman, F. T. Sisco, Frank T. Weems, R. L. Ziegfeld.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

In the following list C/S means change of status; R, reinstatement; M, Member; J, Junior Member; A, Associate Member; S, Student Associate.

Alabama
Leeds—Hohl, Joe L. (M)

Illinois
Chicago—Baumgardner, Hugh S. (A)
Chicago—Knowles, Thomas H. (A)
Chicago—Powell, Donald B. (J)
Chicago—Stuntz, Robert P. (A)
Evanston—Sullivan, James W. (A)
Wilmette—McCandlish, Randolph W. (A)

Indiana
East Chicago—Johnson, Franklin P., Jr. (J)
East Chicago—Mier, Ernest J. (J)
East Chicago—Voreacos, Paul H. (J)
Gary—Van Handel, Ralph A. (A)
Hammond—Absalom, John S. (M) (R. C/S—S-M)

Missouri
Herculaneum—McLellan, James O. (M) (R. C/S—S-M)
St. Louis—Swanson, John S. (M)

New Jersey
Morris Plains—Tocher, Frank L. (A)
Westfield—Rhoda, Richard N. (M)

New York
Little Neck—Richards, John F. (J)
Port Washington—Wemple, Francis H. (A)

Ohio
Youngstown—Kramer, Kenneth H. (M)

Oregon
Grants Pass—Schafer, Max (J)
Portland—Goudge, Carlton E. (A)

Pennsylvania
Fairless Hills—Hanna, Ralph T., Jr. (A)
McKeesport—Walley, Clarence A. (A)
Morrisville—Spitz, Harry S. (M)
Pittsburgh—Brandt, William E. (A)
Pittsburgh—Michels, Norman C. (M)
Pittsburgh—Ow, Charles H., Jr. (M) (C/S—A-M)
Pittsburgh—Tindall, Edwin L. (M)

Texas
Ore City—Kirk, Louis J. (M)

Canada
Toronto, Ontario—Chhabra-Om, P. (J) (R. C/S—S-J)
Quebec (Province), Quebec—Mattoo, Khawaja M. (J)
Montreal—McLaren, Joseph P. (A)

England
Yorkshire, No. Ferriby—Wilmot-Smith, Charles N. (M) (R. C/S—J Foreign Affil.—M)

Japan
Kyushu—Kamekichi, Wada (A)
Tokyo—Tabata, Shintaro (M)

—Coming Events—

Oct. 1-2, Standards Engineers Society, annual meeting, Haddon Hall, Atlantic City, N. J.

Oct. 3-7, Electrochemical Society, Inc., Statler Hotel, Boston, Mass.

Oct. 5, AIME, National Open Hearth Conference, Buffalo Section, Hotel Statler, Buffalo, N. Y.

Oct. 5-9, AIME, Ind. Min. Div., fall meeting, Whiteface Inn, Lake Placid, N. Y.

Oct. 7-8, AIME, National Open Hearth Conference, Southwestern Section, Atlanta, Ga.

Oct. 8, AIME, National Open Hearth Conference, Eastern Section, fall meeting, Warwick Hotel, Philadelphia, Pa.

Oct. 14-16, New Mexico Mining Assn. and El Paso International Mining Assn., joint convention, Carlsbad, N. M.

Oct. 17-20, AIME, Petroleum Branch, Plaza Hotel, San Antonio, Texas.

Oct. 18, AIME, Columbia Local Section, Spokane, Wash.

Oct. 18, AIME, National Open Hearth Conference, Chicago Section, dinner meeting, Phil Smidt's, Chicago, Ill.

Oct. 18-22, National Safety Congress and Exposition, Chicago, Ill.

Oct. 18-22, ASCE, annual meeting, Hotel Statler, New York, N. Y.

Oct. 26, Assn. of Consulting Chemists and Chemical Engineers, Inc., annual symposium and banquet, Hotel Belmont Plaza, N. Y.

Oct. 28-29, ECPD, Hotel Alms, Cincinnati.

Oct. 28-29, AIME, ASME Fuels Conference, William Penn Hotel, Pittsburgh, Pa.

Oct. 29, AIME, NOHC and Pittsburgh Local Section, off-the-record meeting, William Penn Hotel, Pittsburgh, Pa.

Oct. 30, AIME, Utah Local Section, annual fall dinner party, Newhouse Hotel, Salt Lake City, Utah.

Nov. 1-3, AIME, Institute of Metals Div., fall meeting, Hotel Morrison, Chicago, Ill.

Nov. 1-5, American Welding Society, Hotel Sherman, Chicago, Ill.

Nov. 1-5, American Society for Metals, National Metal Congress, Palmer House, exposition, International Amphitheatre, Chicago, Ill.

Nov. 3-5, Steel Founders' Society of America, technical and operating conference, Carter Hotel, Cleveland, Ohio.

Nov. 5-6, AIME, National Open Hearth Conference, Southern Ohio Section, Deshler-Hilton Hotel, Columbus, Ohio.

Nov. 10, AIME, Connecticut Local Section, Hammond Metallurgical Laboratory, Yale University, New Haven, Conn.

Nov. 18-19, National Assn. of Corrosion Engineers, 4th annual conference of western region, Hotel Biltmore, Los Angeles, Calif.

Nov. 28-Dec. 3, ASME, annual meeting, Hotel Statler, New York, N. Y.

Dec. 1-3, AIME, Electric Furnace Steel Conference, William Penn Hotel, Pittsburgh, Pa.

Dec. 12-15, American Institute of Chemical Engineers, annual meeting, Statler Hotel, New York, N. Y.

Dec. 26-31, American Assn. for the Advancement of Science, national meeting, University of California, Berkeley, Calif.

Jan. 13, AIME, Connecticut Local Section, Bridgeport, Conn.

Jan. 23-25, 1955, Indian Institute of Metals, annual general meeting, United Club, Jamshedpur, India.

Feb. 14-17, AIME, annual meeting, Hotel Conrad Hilton, Chicago, Ill.

Mar. 14-15, Steel Founders' Society of America, annual meeting, Drake Hotel, Chicago, Ill.

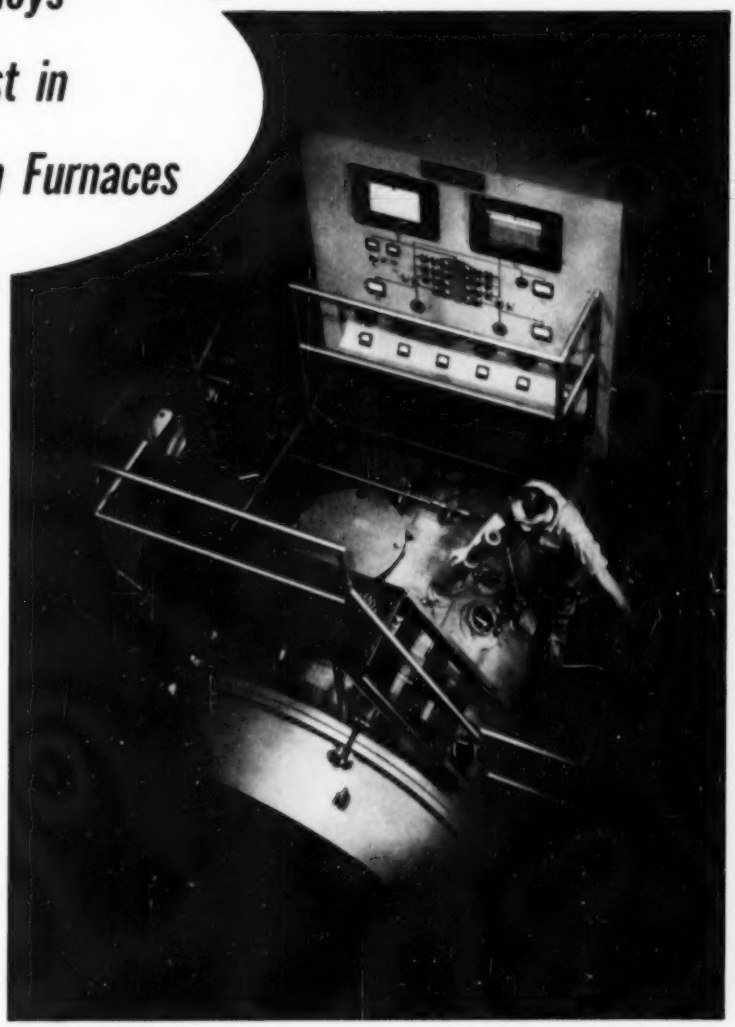
Mar. 16, AIME, Connecticut Local Section, annual meeting, Statler Hotel, Hartford, Conn.

Advertisers' Index

Air Reduction Co., Inc. *G. M. Basford*	1164
Ajax Engineering Corp. *Eldridge, Inc.*	Second Cover
Aluminum Co. of America *Fuller & Smith & Ross Inc.*	1112, 1113
American Bridge Div., U. S. Steel Corp. *Batten, Barton, Durstine & Osborn, Inc.*	*
Bailey Co., Wm. M. *Walker & Downing*	*
Bell Telephone Labs. *N. W. Ayer & Son, Inc.*	1166
Buehler Ltd. *Kreicker & Meloan, Inc.*	1096
Carborundum Co., The *Comstock & Co.*	*
Carondelet Foundry Co. *Whitehead & Sprague, Inc.*	Fourth Cover
Chemical Construction Co. *Michel-Cather, Inc.*	*
Electro Met. Div., Union Carbide & Carbon Corp. *J. M. Mathes, Inc.*	1111
General Electric Co., X-Ray Dept. *Klau, Van Pietersom-Dunlap Assoc.*	1097
General Refractories Co. *Lewis & Gilman Inc.*	*
Great Lakes Carbon Corp. *Davis, Parsons & Strohmeier, Inc.*	*
Harbison-Walker Refractories Co. *Walker & Downing*	1106, 1107
Hevi Duty Electric Co. *Camm & Costigan Inc.*	1103
International Nickel Co. *Marschalk & Pratt Co.*	1114
Metal Hydrides, Inc. *Creamer-Trowbridge Co.*	*
Minneapolis-Honeywell Regulator Co. *Aitkin-Kynett Co., The*	1110
Molybdenum Corp. of America *Smith, Taylor & Jenkins, Inc.*	1108
Morgan Construction Co. *Davis Press, Inc., The*	*
National Carbon Div., Union Carbide & Carbon Corp. *William Esty Co., Inc.*	*
Pittsburgh Lectromelt Furnace Corp. *Fuller & Smith & Ross Inc.*	1098, 1099
Ramtite Co., The *Marsteller, Gebhardt and Reed Inc.*	*
Smeeth-Harwood Co. *Allbright Associates Adv.*	1101
Stokes Machine Co., F. J. *John Mather Lupton Co., Inc.*	Third Cover
Titanium Alloy Mfg. Div., National Lead Co. *Comstock & Co.*	*
U. S. Steel Corp. Columbia-Geneva Steel Div. Tennessee Coal & Iron Div. U. S. Steel Supply Div. U. S. Steel Export Co. *Batten, Barton, Durstine & Osborn, Inc.*	1102A, B, C, D
Vacuum Metals, Inc. *Sutherland-Abbott*	*
Wyman-Gordon Co. *John W. Odlin Co., Inc.*	*

* Previous issue.

High-temperature Alloys now Melted and Cast in Stokes High-Vacuum Furnaces



A Stokes high-vacuum melting furnace of 1000-pound capacity at Utica Drop Forge & Tool Corporation, Utica, N. Y. The furnace is to be used for the melting and casting of high-temperature alloys for jet engine rotor blades.

Vacuum furnace melting and casting is the economical method for producing many new metals, with greatly improved properties. Alloys that can stand up in rocket engine combustion chambers and advanced jet engine turbines, metals essential for the construction of nuclear reactors, still other high-purity metals with properties not previously attainable . . . these are just a few of the more than thirty new elements vacuum processing has added to the industrial spectrum.

Vacuum-melted high alloy steels have greater tensile, yield, and impact strengths than conventionally-processed metal, plus greater stress-rupture strength at elevated temperatures, less creep, less brittleness. High-purity iron, processed in vacuum, has 60 to 75% greater stress-rupture strength and 400% more elongation than conventional metal. In anti-friction bearings, vacuum-processed steel has shown an increase of 300% or more in fatigue strength, and given a whole new perspective to the subject of wear-resistance.

Moreover, vacuum processing of alloys conserves critical hardening elements, since there is minimum loss of these metals during melting. More usable metal is obtained from each melt, and virtually all of the scrap can be salvaged by vacuum melting.

STOKES is *building* vacuum furnaces to process these high-purity metals in quantities up to 2000 pounds, and *planning* 5000-pound units. STOKES vacuum furnaces reflect the practical experience accumulated in fifty years of building vacuum equipment. An interesting NEW brochure is ready for mailing on request!

F. J. STOKES MACHINE COMPANY
PHILADELPHIA 20, PA.

STOKES

STOKES MAKES: High Vacuum Equipment, Vacuum Pumps and Gages / Industrial Tabletting, Powder Metal and Plastics Molding Presses / Pharmaceutical Equipment

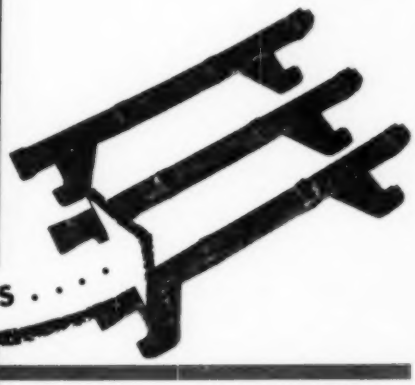
YOUR SINTERING MACHINE Costs may be CUT ...by USING GRATE BARS of 77-NI

1,408 Grate Bars of 77-NI (special alloyed steel) produced by CARONDELET are still serving in the sintering machine shown above in the plant of the Blackwell Zinc Co., Inc., of Blackwell, Okla. This machine has an effective grate area 12 ft. wide and 168 ft. long.

...A HEAT AND ABRASION RESISTANT SPECIAL ALLOYED STEEL GIVING LONGER SERVICE LIFE

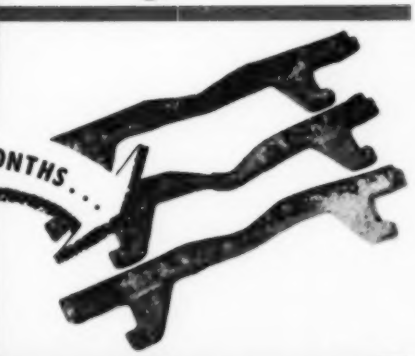
PRODUCED BY **CARONDELET**

Asked to cast Grate Bars that would out-perform ordinary gray or malleable irons in overcoming burning, warpage and abrasion, CARONDELET produced those shown herein of 77-NI using CARONDELET'S Electric Furnace process for metallurgical control.



STILL IN SERVICE AFTER 2½ YEARS....

Still in use after 2½ years, the above Grate Bars represent an extended service life that promises to reach approximately five years! The ratio of their service life to usual grate bar materials ranges from 6 to 1 to 10 to 1 as proved by this demonstrated service and others in non-ferrous sintering.



SPECIAL GRAY IRON GRATE BARS LASTED LESS THAN 6 MONTHS...

Alloyed heat-resistant gray iron Grate Bars used concurrently to test a modified design, lasted less than 6 Months.

SEND FOR DESCRIPTIVE BOOKLET

...THAT PRESENTS IN DETAIL THE OPERATION AT CARONDELET TO THE BENEFIT OF VARIED INDUSTRY

CARONDELET FOUNDRY CO.

2101 S. KINGSHIGHWAY BLVD.
ST. LOUIS 10, MISSOURI